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PRACTICAL

Test-Book of Chemistry

BY  
JOHN DABNEY PALMER, M.A., M.D.

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FIRST THOUSAND



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BY

**JOHN DABNEY PALMER**

## PREFACE.

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IN collecting these Tests and arranging them alphabetically, an effort is made to supply physicians engaged in practice, and pharmacists, with a ready means and safe guide for testing any substance presented for examination. And as only practical information is aimed at, the methods employed are very simple, and form a series of unpretentious experiments.

The Tests have been garnered from every available source, such as standard works on chemistry, "Proceedings of the American Pharmaceutical Association," and pharmaceutical journals; and in many instances are copied into the collection without undergoing any change. It is a recognized duty to give all authors credit for their work, but the plan and limits of this Test-book forbid particular citation of the many works consulted in preparing it; it is a book made from other books.

The spelling adopted for scientific and chemical words is that laid down by the rules of the American Association for the Advancement of Science, 1891. The temperatures are given in Centigrade.

J. D. P.

April, 1907.

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## SPECIFIC TESTS.

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### **Test for Acetanilid—Phenylacetamid—Antifebrin,** $\text{C}_6\text{H}_5\cdot\text{NH}(\text{C}_2\text{H}_3\text{O})$ .

Acetanilid is obtained by the action of glacial acetic acid on aniline. It is in white, shining, crystalline scales, or a crystalline powder, odorless, with a greasy feel, and a slight burning taste. Soluble in 200 parts of water at  $15^\circ\text{C}$ ., in 18 parts of boiling water; easily soluble in alcohol, ether, chloroform, and benzine; very little in carbon disulphid; melting-point  $114^\circ\text{C}$ . The solution is neutral to litmus paper.

(1) 0.10 gm. ( $1\frac{1}{2}$  grs.) Acetanilid heated with 1 c.c. (16 minims) of sodium hydrate solution (Reagent 20) and three drops of chloroform gives off the offensive poisonous phenylisocyanid,  $\text{C}_6\text{H}_5\text{NC}$  (the isonitril reaction); the mixture becomes yellowish red by adding bromin-water (Reagent 2).

(2) 0.10 gm. ( $1\frac{1}{2}$  grs.) Acetanilid is soluble in 1 c.c. (16 minims) of sulfuric acid (Reagent 21). The solution remains colorless and clear after the addition of 3 c.c. (48 minims) of water, but in an hour crystals of Acetanilid begin to form; sooner, when 0.20 gm. (3 grs.) Acetanilid has been taken for the experiment.

(3) If 0.10 gm. ( $1\frac{1}{2}$  grs.) Acetanilid is heated to boiling with 1 c.c. (16 minims) of hydrochloric acid (Reagent 5),

an equal volume of a saturated watery solution of phenol, and also 1 c.c. (16 minims) of a saturated watery solution of chlorinated lime is added,  $\text{Ca}(\text{OCl})_2$ , a turbid red or violet fluid is obtained, turning dark blue on adding ammonia-water (Reagent 1) in excess (indophenol reaction).

(4) Triturate 0.032 gm. ( $\frac{1}{2}$  gr.) Acetanilid with an equal amount of sodium nitrate or nitrite, and strew upon it 1 c.c. (16 minims) of sulfuric acid (Reagent 21); a bright-red-colored solution is produced.

(5) Dissolve 0.032 gm. ( $\frac{1}{2}$  gr.) potassium dichromate in 10 c.c. (162 minims) of water, and add to the solution 30 gm. (1 oz., 25 grs.) of concentrated sulfuric acid (Reagent 21). Shake 0.10 gm. ( $1\frac{1}{2}$  grs.) of Acetanilid with 1 c.c. (16 minims) of this solution; an intense-red color, immediately turning blue and soon fading, is developed.

(6) 0.10 gm. ( $1\frac{1}{2}$  grs.) Acetanilid heated with 2 c.c. (32 minims) sulfuric acid (Reagent 21) until light brown, cooled off, and mixed, a little at a time, with bromin-water (Reagent 2) gives a very light-red-colored fluid. Very soon an abundance of white, microscopically small crystal needles appear.

(7) 0.20 gm. (3 grs.) Acetanilid is not changed by 2 c.c. (32 minims) of nitric acid (Reagent 15). By warming it is dissolved, whereby a yellowish-red-colored fluid is obtained; heating further to the boiling-point does not change the color. When the test-tube is kept for hours in the water-bath, however, the hue changes to a dark-brown red.

(8) Boil a small quantity of Acetanilid in hydrochloric acid (Reagent 5); it is decomposed into aniline and acetic acid.

(9) Heat Acetanilid with an equal amount of dry zinc chlorid to  $250^{\circ}\text{C}$ .; it yields aromatic vapors, which color wood-shavings yellow, and flavanilin, a dyestuff, which

dissolves in hydrochloric acid with a moss-green color. The residue is also yellow.

(10) Boil 0.50 gm. (8 grs.) of Acetanilid in 8 c.c. (130 minims) of water. Let it cool, filter, and add to the filtrate a few drops of dilute nitric acid in which a little potassium nitrite is dissolved; then a few drops of nitroso-nitric acid, and boil again. A distinct red color appears (differing in this respect from phenacetin, which undergoes no change).

(11) Dissolve 1 gm. ( $15\frac{1}{2}$  grs.) Acetanilid in 6 c.c. (100 minims) of chloroform and add 20 c.c. (325 minims) of petroleum ether; a precipitate forms after standing a short time (differing in this respect from exalgin).

(12) Mix a saturated aqueous solution of Acetanilid with half its volume of bromin-water (Reagent 2); it is colored red immediately, and in a few minutes a crystalline precipitate appears (differing in this respect from phenacetin).

(13) Add a small quantity of Acetanilid to a boiling solution of potassium permanganate; it is not altered and does not reduce the permanganate (absence of acetoluid).

(14) Boil 0.10 gm. ( $1\frac{1}{2}$  grs.) Acetanilid in 1 c.c. (16 minims) of potassium-hydrate solution, 1:4, and suspend a drop of filtered solution of chlorinated lime on a glass rod over the hot solution; the drop is soon colored yellow, with a violet tinge by reflected light; upon continued heating it turns violet.

(15) Add Acetanilid to a solution of potassium bismuthous iodid; a precipitate is thrown down.

Solution of potassium bismuthous iodid is made by mixing 43 c.c. (1 f. oz., 211 minims) liquor bismuthi, Br, with 9 gm. (139 grs.) of potassium iodid and 9 c.c. (146 minims) of strong hydrochloric acid (Reagent 5).

(16) Acetanilid in urine is found by shaking the urine

with chloroform, evaporating, and heating the residue with mercurous nitrate; a green color develops if Acetanilid is present.

(17) If 0.065 gm. (1 gr.) Acetanilid be agitated for one-half hour with 200 c.c. (6 f. oz., 366 minims) of distilled water at the ordinary temperature, a clear solution will result (absence of phenacetin).

(18) On heating 0.1 gm. ( $1\frac{1}{2}$  grs.) of Acetanilid with 10 c.c. (162 minims) of water, filtering the solution when cold, and adding bromine T.S., drop by drop, to the filtrate, a whitish precipitate of parabromacetanilid is formed (distinction from antipyrin or acetphenetidin).

(19) A cold, saturated, aqueous solution of Acetanilid, added to ferric chlorid T.S., should not affect the color of the latter (absence of aniline salts and various allied substances).

(20) To detect Acetanilid when mixed with phenacetin, boil 0.1 gm. ( $1\frac{1}{2}$  grs.) of the substance to be tested, for one minute, with 1 c.c. (16 minims) of concentrated hydrochloric acid (Reagent 5), and dilute with 10 c.c. (162 minims) of water; filter off the precipitate obtained (pure Acetanilid gives little or no precipitate), and to the filtrate add three drops of a 3 per cent solution of chromic acid. When the test is thus applied, pure phenacetin gives a permanent ruby-red color. Pure Acetanilid gives first a pure yellow color, quickly becoming greenish yellow, then green, later very dark green, and after standing some time, a green flocculent precipitate separates.

(21) If a 1 per cent solution of sodium vanadate be substituted for the chromic acid in (20), essentially the same results are obtained. Pure phenacetin in this case gives a yellow color, quickly becoming violet, then purple, changing to very deep purplish red. This color is not permanent, however, and after standing some hours fades to a very light reddish violet. Pure Acetanilid gives a yellow

coloration at first, almost instantly changing to greenish yellow, then a greenish-blue fluorescence appears, the green gradually growing stronger, until finally after a few hours a beautiful green precipitate settles out, leaving the solution colorless.

(22) On boiling Acetanilid with concentrated hydrochloric acid (Reagent 5) a slightly greenish solution results. On diluting with water the solution gives no reaction with chromic-acid mixture, but on the addition of one or two drops of fuming nitric acid a handsome green color, changing to deep blue, develops within two or three minutes.

(23) Acetanilid may be distinguished from phenacetin by boiling with aqueous phenolglycerin solution, cooling, and adding an excess of a filtered bleaching-powder solution. Acetanilid gives a brownish coloration, changing shortly to an indigo-blue, while phenacetin gives a flocculent, light, coffee-colored precipitate.

### Test for Acetone, $\text{CH}_3 \cdot \text{CO} \cdot \text{CH}_3$ .

Acetone is obtained by the distillation of certain acetates, or by the destructive distillation of citric acid, starch, sugar, or gum, with quicklime. It is a transparent, colorless, mobile, and volatile liquid, with a pleasant ethereal odor and a pungent, sweetish taste. Specific gravity 0.790 at 25° C. Miscible with water in all proportions, without cloudiness; also miscible with alcohol, ether, chloroform, and volatile oils. It volatilizes at low temperatures and boils at 56.5° C. It is inflammable and burns with a luminous non-sooty flame. It is neutral to litmus paper previously moistened with water.

(1) If 20 c.c. (324 minims) of Acetone contained in a clean, glass-stoppered vial be mixed with 0.1 c.c. (2 minims) of tenth-normal potassium permanganate T.S., the

pink tint produced by the admixture should not wholly disappear in less than 15 minutes (limit of *empyreumatic substances*).

(2) To detect Acetone in urine, add to the urine a few drops of a concentrated solution of nitro-prussiate of soda and make the solution alkaline by adding potash. A red coloration appears and then goes off; add acetic acid, and if Acetone be present, we get a dark-violet color.

(3) A trace of Acetone may be detected by the addition of a solution of iodine in potassium iodide along with an alkali, when iodoform is obtained, the odor and color of which are distinctive.

(4) Acidulate the aqueous solution with a few drops of phosphoric acid, and then add small quantities of copper sulfate solution and solution of iodine in potassium iodide; if Acetone is present a brown cloudiness appears; on heating, the liquid is decolorized and a grayish-white pulverulent, voluminous precipitate occurs which contains iodine and copper in organic combination. The precipitate is almost insoluble in water. The reaction is very sensitive.

(5) Dissolve 0.5 gm. ( $7\frac{1}{2}$  grs.) potassium iodide in 50 c.c. (1 f. oz., 331 minims) distilled water, add 1 gm. ( $15\frac{1}{2}$  grs.) iodine, filter, and then add to the filtrate 5 gm. (77 grs.) methylamine. In an alkaline Acetone solution this reagent gives rise to the formation of iodoform, which immediately unites with the nitrogen of the amine, giving rise to the formation of an isonitrile which is easily recognized by its very characteristic odor. In applying the test, the absence from the urine of alcohol, chloroform, and lactic acid must first be verified. The urine is then filtered and 10 c.c. (162 minims) rendered alkaline by the addition of 1 c.c. (16 minims) of a 1:10 soda-lye solution; 1 c.c. (16 minims) of the reagent is now added

and the whole boiled. If Acetone is present, the highly disagreeable odor of isonitrile develops.

**Test for Acetphenetidin,  $C_{10}H_{13}NO_2$ .**

Acetphenetidin, a derivative of phenol, is in white, glistening, crystalline scales, or in a fine crystalline powder, odorless, and tasteless; soluble in 925 parts of cold water; in 70 parts of boiling water; soluble in alcohol, ether, and chloroform. It dissolves in sulfuric acid (Reagent 21) without color; but if shaken with nitric acid (Reagent 15) it is colored yellow, which color persists when heated.

(1) If 0.10 gm. ( $1\frac{1}{2}$  grs.) of Acetphenetidin be boiled for one minute with 1 c.c. (16 minims) of concentrated hydrochloric acid (Reagent 5) and the solution diluted with 10 c.c. (162 minims) of water, cooled, and filtered, it should yield on the addition of three drops of an aqueous solution of chromium trioxid (1:30) a ruby-red color.

(2) If 0.10 gm. ( $1\frac{1}{2}$  grs.) Acetphenetidin be boiled with 10 c.c. (162 minims) of water, it should yield a solution which, when cooled and filtered, should not become turbid upon the addition of bromin T.S. in slight excess (absence of *acetanilid*).

(3) If 0.10 gm. ( $1\frac{1}{2}$  grs.) of Acetphenetidin be boiled for one minute with 3 c.c. (48 minims) of sodium hydrate solution (Reagent 20), the solution cooled and agitated with 5 c.c. (81 minims) of solution of chlorinated soda, there should be produced a clear yellow liquid, and not a purplish-red or brownish-red cloudy liquid or precipitate (absence of *acetanilid*).

**Test for Aconitin,  $C_{33}H_{45}NO_{12}$ .**

Aconitin is in a white, amorphous, or crystalline powder, with an intensely bitter taste, followed by a tingling sensation and prolonged numbness; soluble in alcohol, ether,

chloroform, acetic acid, and benzene; sparingly soluble in cold, more freely in hot water; insoluble in carbon disulfid and glycerin. A cold watery solution of it has an alkaline reaction on litmus paper or towards tincture of cochineal (1:10).

(1) Dissolve 0.010 gm. ( $\frac{1}{10}$  gr.) of Aconitin, or one of its salts, in 1 c.c. (16 minims) of nitric acid (Reagent 15), evaporate to dryness, add to the residue, when cool, a few drops of an alcoholic solution of caustic potash (1:5), the odor of benzoic ethyl ester is developed.

(2) Dissolve 0.10 gm. ( $1\frac{1}{2}$  grs.) Aconitin in 250 c.c. (8 f. oz.) of water by the aid of alcohol and a drop of acid. Touch the tongue with a drop of the solution; a tingling sensation, suggesting a feeling of having been burned, followed by numbness, is experienced.

(3) To 5 c.c. (81 minims) of the above (2) solution add a few drops of a 5 per cent solution of gold chlorid ( $\text{AuCl}_3$ ); a voluminous yellowish precipitate is obtained.

(4) To 5 c.c. (81 minims) of the (2) solution add 0.06 c.c. (1 minim) solution of platinic chlorid; a white precipitate is formed.

(5) To 5 c.c. (81 minims) of the (2) solution add 20 c.c. (324 minims) water and a few drops of mercuric potassium iodid solution (Reagent 12); an opalescence only is formed.

(6) Dissolve 1 gm. ( $15\frac{1}{2}$  grs.) of iodin and 2 gm. (31 grs.) of potassium iodid in 125 c.c. (4 f. oz.) of water and add a few drops of the solution to a small quantity of the (2) solution; a voluminous yellowish-brown precipitate is obtained.

(7) Mix 0.005 gm. ( $\frac{1}{12}$  gr.) of amorphous Aconitin with 0.010 gm. ( $\frac{1}{10}$  gr.) sugar and two drops of sulfuric acid (Reagent 21); a pink, sometimes yellowish-red color is produced.

(8) Aconitin, in fluid extract of aconite, is tested by adding ammonia-water (Reagent 1) to 5 c.c. (81 minims)

of the fluid extract until alkaline, then exhausting it with ether. Evaporate the ether; add 0.5 c.c. (8 minims) of water to the residue; warm; filter, and add a few drops of the filtrate to 2 c.c. (32 minims) of Reagent 23; a purple hue appears, disappearing in a few seconds.

(9) To a small quantity of the (2) solution add a few drops of solution of potassium or sodium hydrate; it is precipitated, but no precipitate is formed by adding potassium or sodium bicarbonate or ammonium carbonate.

(10) Dissolve 0.10 gm. ( $1\frac{1}{2}$  grs.) Aconitin in dilute phosphoric acid and evaporate slowly; when at a certain degree of concentration a violet coloration appears.

(11) Sprinkle a small quantity of Aconitin on a drop of colorless concentrated sulfuric acid (Reagent 21); the acid turns yellowish and then a dirty violet color.

(12) Add 0.10 gm. ( $1\frac{1}{2}$  grs.) Aconitin to 2 c.c. (32 minims) of Reagent 25; a yellowish-brown color is produced.

(13) Add 0.06 or 0.12 c.c. (1 or 2 minims) ferric-chlorid solution (Reagent 4) to 0.30 c.c. (5 minims) of a watery solution of Aconitin; a yellow precipitate is formed.

(14) Aconitin, when dropped upon sulfuric or nitric acid, should produce no color, but if rubbed with sulfuric acid (Reagent 21) containing a crystal of ammonium vanadate an orange color is produced.

(15) On evaporating 0.01 gm. ( $\frac{1}{8}$  gr.) of Aconitin with 5 drops of fuming nitric acid, the resulting yellow residue, when cooled, should not yield a violet color when treated with alcoholic potassium hydroxide T.S. (difference from pseudaconitin and atropin).

(16) Any soluble salt of Aconitin in dilutions of 1 in 1000 produces, with a drop of potassium permanganate T.S., a blood-red precipitate of Aconitin permanganate.

### Test for Æsculin, $C_{15}H_{16}O_9 + 3H_2O$ .

Æsculin is a glucoside obtained from the *Æsculus hippocastanum*, or horse-chestnut. It crystallizes in small white prisms having a bitter taste and acid reaction. It is readily soluble in hydrochloric acid, very sparingly soluble even in warm carbon disulfid, and insoluble in warm and in cold chloroform. It is highly fluorescent, displaying a fine blue hue.

(1) Add to a small particle of Æsculin two or three drops of concentrated sulfuric acid (Reagent 21); it is quickly charred.

(2) Boil a few crystals of Æsculin in a test-tube with dilute hydrochloric acid; it is decomposed into glucose and æsculetin (dioxycoumarin).

### Test for Albumen in Urine.

The presence of Albumen in the urine is ascertained by a very simple process, which, if carefully performed, leads to positive results. The reaction of clear or previously filtered urine is first tried, and a test-tube half full of it is then heated over a spirit-lamp. If it contains Albumen and has an acid reaction, the surface of the urine becomes turbid when the heat exceeds  $70^{\circ} C.$ , and coagulation of the Albumen quickly follows. If the urine be either neutral or alkaline, the coagulation will not take place, or there will be at most only a milky turbidness. But if, before heating, we add to the urine a little acetic acid, carefully avoiding an excess, a flaky coagulation will take place in the urine when boiled. If, again, the urine be very acid, and contains, for instance, free hydrochloric or nitric acid, which may readily happen when these acids have been taken internally, boiling may fail to produce coagulation of the Albumen. To obtain the Albumen in

such case the urine must be, first of all, sufficiently neutralized with very dilute ammonia. When all these precautions have been taken, if we obtain, on boiling the urine, a turbidity or precipitate which on cooling is not dissolved by nitric acid, we may consider the presence of Albumen in it demonstrated.

Cases, however, are occasionally met with in which a precipitate is formed on boiling the urine (particularly if the urine is only slightly acid or neutral), but in which, nevertheless, no trace of Albumen is present. Such precipitate consists of phosphatic earths, which in slightly acid urine are generally held in solution by the free carbonic acid. On the expulsion of the gas by boiling, the phosphates are precipitated in a flocculent form, and can then scarcely be distinguished by the eye from coagulated Albumen. All doubt as to its nature is immediately removed by the addition of a few drops of dilute hydrochloric acid to the urine (when cooled) in which the precipitate is suspended. If the precipitate consists of phosphates it, will immediately disappear and leave the fluid clear, but if of Albumen it will remain unchanged.

(1) Albumen is completely dissolved when exposed to the action of a solution of caustic potash or soda; the solution has a yellowish color and contains Albumen in an altered state. On neutralizing the alkali with an acid, the dissolved Albumen is thrown down and sulfuretted hydrogen is evolved.

(2) Concentrated acetic acid assisted by heat dissolves Albumen; and in this solution potassium ferrocyanide ( $\text{K}_4\text{Fe}(\text{CN})_6 + 3\text{H}_2\text{O}$ ) and potassium ferricyanide ( $\text{K}_6\text{Fe}_2(\text{CN})_{12}$ ) throw down peculiar precipitates.

(3) A violet-colored fluid is produced when Albumen is heated with concentrated hydrochloric acid (Reagent 5), and better still, with the addition of a little sulfuric acid (Reagent 21).

(4) Concentrated nitric acid (Reagent 15) colors it yellow (xanthoproteic acid) when heated.

(5) A solution of 1 part of mercury in 2 parts of nitric acid (Reagent 15) containing four and a half equivalents of water (sp. gr. 1.41) is the most delicate test for Albumen, as well as for all protein bodies whether dissolved or undissolved. An albuminous fluid heated with this solution of mercury to from 60° to 100° C. becomes of an intensely red color, which does not disappear on exposure to the air or after long boiling.

(6) Albumen becomes a brownish yellow when a solution of iodine in hydriodic acid is added to it. This reaction is especially well observed under the microscope.

(7) Heated on platinum foil, Albumen rapidly becomes brown, swells up and gives off an odor like that of burnt horn. The bulky carbonaceous mass remaining is burned with difficulty and leaves a grayish ash consisting chiefly of lime and phosphoric acid.

(8) Urine containing Albumen heated in a test-tube over a spirit-lamp becomes turbid at a temperature of about 75° to 80° C. The coagulation commences at the surface of the fluid and then extends gradually downwards through the tube. A white flocculent coagulum, which, under certain circumstances, is more or less colored, is thus formed, the Albumen passing into an insoluble form. Several points are worthy of notice in reference to this simple test: When the solution of Albumen is very much diluted, the turbidity will often appear only at a boiling heat; and to obtain a distinct coagulum, it may be necessary to boil it for a long time and then allow it to stand. When the urine has a slightly acid reaction, and provided there be no excess of acid present, complete coagulation usually occurs. But if the solution be neutral or slightly alkaline, heat often occasions merely a slight turbidity, even though a considerable quantity of Albumen is present.

In such case the Albumen remains in solution with the alkali. If, however, before heating the urine, acetic acid is added to neutralize the alkali, the Albumen is completely coagulated and thrown down in large flocculi. Care should be taken not to add an excess of the acid, because Albumen is more or less dissolved by free acetic acid during boiling, and also by hydrochloric acid, the latter giving it a reddish-blue color.

(9) Dilute nitric acid added to a solution of Albumen throws down a white precipitate of Albumen nitrate, which is soluble in a large quantity of water, an important fact. Other mineral acids produce a like effect.

(10) Strong alcohol produces complete coagulation of Albumen in solution. Dilute alcohol occasions a precipitate, but does not convert the Albumen into its insoluble form.

(11) Most of the metallic salts, and likewise alum, occasion differently constituted precipitates in urine containing Albumen.

(12) Sugar and concentrated sulfuric acid (Reagent 21) become of a beautiful red color with Albumen and all protein bodies.

(13) Albumen treated with a solution of copper sulfate and heated after the addition of caustic soda or potash imparts to the solution a beautiful violet color. This reaction does not take place, or at all events is imperfect, if the alkali is added before the salt of copper.

(14) A little pure concentrated nitric acid (Reagent 15) is poured into a champagne-glass, and the urine to be tested then allowed, by means of a pipette, to run down the side of the glass and spread over the surface of the acid. When this is carefully done, the urine floats on the surface of the acid and their admixture takes place slowly and gradually. In most cases an intensely red, violet, or blue ring (the reaction of uroxanthine) presents

itself at the point of contact of the two fluids. If the urine contains mere traces of Albumen, this test exhibits at the part where the fluids come in contact a circular turbidity well defined above and below. This reaction lasts for some length of time, but the coagulated Albumen at last sinks to the bottom of the glass. This is Heller's test. A quick and handy method of performing this test, useful when the amount of urine is small, or when there are many examinations to be made, as in hospitals or dispensaries, is to dip a pipette of  $\frac{1}{4}$ -in. caliber into the urine, taking up about 1 c.c. (16 minims), and then dipping the same into nitric acid, relaxing the finger pressure so as to admit the acid. A sharp white belt is formed at the line of contact if it contains Albumen.

(15) To five volumes of cold *saturated* solution of magnesium sulfate add one volume of nitric acid (sp. gr. 1.42) and preserve this reagent for use. Pour some perfectly clear filtered urine into a test-tube and carefully add an equal volume of the reagent, delivered gently from a pipette, so that the liquids shall not mix. An opalescent zone will form at the point of contact either immediately or within twenty minutes, according to the quantity of Albumen present. This zone should not dissolve on gently warming, but should be a distinct ring at the bottom of the urine, and not a general haze near the top, which latter indicates *mucin*. If the zone of contact has a pink color, *indican* or other coloring-matter is excessive.

(16) Nearly fill a long test-tube with clear urine (filtered if necessary) and add one or two drops of acetic acid; then, holding the test-tube by its lower end, boil the upper portion of the urine; a cloudiness in the boiled portion, which, on addition of a few drops of acetic acid, does not disappear, indicates the presence of Albumen. The cloudiness is seen more distinctly by holding the test-tube in front of a black coat-sleeve.

(17) Pour 10 c.c. (162 minims) of a saturated solution of picric acid into a test-tube and add to it, drop by drop, the suspected urine, previously acidified with a drop of acetic acid; a cloudiness is produced if Albumen is present. If the test is added to the urine no reaction takes place.

(18) Acidulate a drachm of clear urine in a test-tube with acetic acid and add potassium-ferrocyanide solution, drop by drop, until an excess has been added; if a cloudiness or precipitate forms, Albumen is to be suspected; a mere trace requires some time to form the cloud. This is Bödeker's test.

(19) Add to the urine in a test-tube a few drops of acetic acid and a small quantity of sodium chloride (common salt) and heat it; Albumen, if present, is completely precipitated.

(20) Dissolve 3.24 gm. (50 grs.) potassium chloride and 1.35 gm. (21 grs.) mercuric chloride in 20 c.c. (324 minims) of acetic acid; dilute with water to 60 c.c. (2 f. oz.) and add it to the urine; a white precipitate, insoluble in acetic acid, indicates the presence of Albumen. Peptones cause a precipitate which redissolves on boiling.

(21) Mix equal volumes of carbolic and glacial acetic acids and add the mixture, a little at a time, to the urine; a white precipitate forms if Albumen is present.

(22) Add to the urine fifteen drops of alcohol, followed by fifteen drops of carbolic acid; a precipitate indicates Albumen.

(23) Mix 1 part of picric acid, 2 parts of citric acid, 50 parts of water, and 30 parts of alcohol; dissolve and add water to 100 parts. Add this, drop by drop, to the clear urine; a precipitate indicates Albumen.

(24) Mix 1 part of tartaric acid, 5 parts of mercuric chloride (corrosive sublimate), 10 parts of sodium chloride

(common salt) with 100 parts of distilled water, and add 5 parts of 40% formaldehyde. Put 2 c.c. (32 minims) of this in a test-tube and overlay it with 3 or 4 c.c. (48 or 65 minims) of urine; a ring appears immediately at the line of separation of the liquids if Albumen is present. This test is said to be sensitive to 1 part of Albumen in 370,000 parts of urine.

(25) Heat 4 or 5 c.c. (65 or 81 minims) of non-alkaline urine in a test-tube to near the boiling-point; then stop heating and add a few drops of strong formaldehyde. If the urine contains any Albumen, it coagulates, the flocculent mass collects on the surface of the urine and adheres to the walls of the tube. When the urine is strongly albuminous, the reaction is given without heat.

(26) Acidulate the urine with acid phosphate of soda; allow it to settle; filter, to remove mucus and urates. Then pour it in a test-glass and mix with dilute carbolic acid (1:20); a turbidity, followed by a flocculent deposit, is observed even when very small quantities of Albumen are present.

(27) Mix 10 c.c. (162 minims) of a 10 per cent solution of potassium sulphocyanate (KSCN) with 2 c.c. (32 minims) of acetic acid; add a few drops of this liquid to the urine; a cloudiness or precipitate indicates Albumen.

(28) Dissolve 1 part mercuric chloride (corrosive sublimate) in 20 parts distilled water, and then 1 part potassium iodid in 2 parts distilled water; mix the two solutions; dip strips of filtering-paper into the mixture and let them dry; acidulate the urine with a few drops of acetic acid and dip a strip of the paper into it; an immediate precipitate indicates Albumen.

(29) Add, drop by drop, to 5 or 6 c.c. (81 or 97 minims) of previously filtered urine, a (1:10) solution of chromic acid; the immediate appearance of a white flocculent sediment renders the presence of Albumen highly prob-

able; and if it remains, on heating, its presence is positively demonstrated. The chromic-acid reaction is very sensitive; it permits the detection of 1 part of Albumen in 50,000 parts of urine.

(30) Add to a test-tube containing water an amount of resorcin equal to one-third of the water in the tube; when dissolved, allow the urine to flow gently down the tube on the solution; a ring is formed which does not disappear on heating if Albumen is present.

(31) Half fill a test-tube with clear urine; add to it, a little at a time, fuming hydrochloric acid; heat it; a violet color is developed if it contains Albumen.

(32) A few drops of a saturated solution of salicyl-sulphonic acid ( $C_6H_3(OH)_3 \cdot SO_3H \cdot COOH$ ) are added to 20 or 30 minims of the urine in a small test-tube. If no precipitate is formed, there is no Albumen or body present. If there is a precipitate, the urine is boiled, and the Albumen becomes coagulated and flaky. This reagent is very sensitive and precipitates only protein bodies.

(33) A solution of bichloride of mercury dropped into urine containing Albumen causes a white precipitate. Bostock's test.

(34) Tannin or tincture of galls gives a yellow, pitchy precipitate in albuminous urine.

(35) If a small splinter of the glassy metaphosphoric acid ( $HPO_3$ ) is shaken a few moments with a little water, and the solution added to the urine, the Albumen present is at once precipitated in flakes. It is important that the solution of metaphosphoric acid be prepared immediately before use.

(36) Add to the urine an equal amount of an alcoholic solution of tannin, 1.5 gm. (23 grs.) in 100 c.c. (3 f. oz., 183 minims) of 90 per cent alcohol. The whole is then heated and an equal quantity of a 33 per cent aqueous

solution of hydrochloric acid is added equal to the quantity of urine. In the presence of Albumen the fluid becomes opaque and the Albumen is gradually thrown down in a yellowish-white precipitate. This test will detect 1 part of Albumen in 200,000 parts of urine.

(37) 50 c.c. (1 f. oz., 331 minims) of a saturated solution of magnesium sulfate are added to a mixture containing 2 gm. (31 grs.) of corrosive sublimate, 4 gm. (62 grs.) of succinic acid, 4 gm. (62 grs.) of common salt, and 50 c.c. (1 f. oz., 331 minims) of water. The urine is first acidified by adding 1 c.c. (16 minims) of acetic acid to 5 c.c. (81 minims) of urine, the liquid being then filtered. Then, by the aid of a pipette, the filtered urine is slowly floated on the surface of the reagent in an inclined test-tube. If a ring of Albumen appears, then heat. The Albumen will not disappear.

### **Test for Aloin, $C_{16}H_{18}O_7$ .**

Aloin is a lemon-yellow or dark-yellow crystalline powder possessing a slight odor of aloes and an intensely bitter taste. Soluble in 65 parts of cold water, freely in hot water; in 11 parts alcohol; in acetone; slightly soluble in ether, chloroform, and benzene; the watery solution is yellow; turns brown on standing; is neutral to litmus paper.

(1) Ammonia-water (Reagent 1) and alkali solutions dissolve Aloin, forming a yellow solution soon turning red and exhibiting a greenish-red fluorescence.

(2) Aloin added in minute quantity to concentrated sulfuric acid (Reagent 21) forms a yellowish-red solution which, upon the addition of a small crystal of potassium dichromate ( $K_2Cr_2O_7$ ), changes to an olive-green, then to a dark green, and finally, on standing, to a blue. If a large amount of potassium dichromate ( $K_2Cr_2O_7$ ) be used,

the yellowish-red solution first turns purple, then brown, and finally green.

(3) Bromin-water (Reagent 2) added to the aqueous solution of Aloin produces a pink color.

(4) Gold chlorid T.S., when added to an aqueous solution of Aloin, turns it carmine-red, changing later to violet.

(5) A drop of ferric-chlorid solution (Reagent 4) added to an alcoholic solution of Aloin produces a brownish-green color.

(6) If to a dilute aqueous solution of Aloin obtained from Curacao aloes 1 drop of copper sulfate T.S. be added, a bright-yellow color will be produced; upon adding a few drops of concentrated solution of sodium chloride the liquid will acquire a red color; and upon further addition of a little alcohol, the color will be changed to violet (distinction from *Nataloin* and *Capaloin*).

### Test for Alum, $\text{Al}_2\text{SO}_4$ , $\text{K}_2\text{SO}_4$ , $24\text{H}_2\text{O}$ .

Alum is in large colorless octahedral crystals, sometimes modified by cubes, or in crystalline fragments, without odor, but having a sweetish and strongly astringent taste. It is soluble in 9 parts of water at  $15^\circ\text{C}$ ., and in 0.3 part of boiling water; it is also freely soluble in warm glycerin, but is insoluble in alcohol. It has an acid reaction.

(1) To a solution of Alum add ammonium sulphhydrate ( $\text{NH}_4\text{HS}$ ); a gelatinous white precipitate (aluminium hydrate) falls.

(2) To solution of Alum add ammonia: aluminium hydrate falls; add excess of ammonia: the precipitate is practically insoluble.

(3) To the solution of Alum add solution of potash: again aluminium hydrate falls; add excess of potash and agitate: the precipitate dissolves.

(4) Acidify a portion of the alkaline solution (3) with hydrochloric acid, add ammonia *slightly* in excess and apply heat. A white flocculent precipitate insoluble in more ammonium chloride indicates *aluminium*.

(5) The most convenient test for Alum in *bread* is made with a freshly prepared tincture of logwood. This tincture is made by digesting 5 gm. (77 grs.) of freshly cut logwood chips with 100 c.c. (3 f. oz) of alcohol. Having diluted 5 c.c. (81 minims) of the logwood tincture with 90 c.c. (3 f. oz.) of water and added 5 c.c. (81 minims) of saturated solution of ammonium carbonate, the mixture is *immediately* poured over 10 gm. (154 grs.) of bread in a glass dish. After five minutes the liquid is poured off, the bread slightly washed and dried at 100° C. A lavender or dark-blue color denotes the presence of Alum. Pure bread is at first reddish, fading to a yellow or light brown.

(6) Alum in drinking-water may be detected by adding to the suspected water enough fresh tincture of logwood (5) to give a decided color, then a solution of ammonium carbonate. If a blue precipitate falls, then Alum is present at least 1:1000; if no precipitate, but a blue color persists for one hour, then Alum is present at least 1:50,000. If before the hour be out the color be brown or pink, then there is no Alum.

(7) To detect Alum in red wine boil a sample of the wine for a few minutes; pure wine remains unchanged, while the adulterated article becomes turbid.

### Test for Ammonia, $\text{NH}_3$ .

Ammonia is a colorless gas; of an exceedingly pungent suffocating odor; caustic taste; alkaline reaction; lighter than air; very soluble in water, which will absorb 700 times its volume at ordinary temperatures and thereby acquire the properties of the gas itself; soluble in alcohol,

ether, glycerin, and essential oils; absorbed by charcoal, sugar, sponge, cloth, and all porous bodies.

(1) Ammonia is recognized by its odor; by bringing a glass rod dipped into hydrochloric acid near it, which will produce dense, white fumes of ammonium chlorid ( $\text{NH}_4$ ); or by holding in the mouth of a test-tube containing it a strip of moistened red litmus paper, which will be immediately turned blue by the alkaline vapor, in which form the ammonia ( $\text{NH}_3$ ) exists.

(2) To a solution of a salt of ammonium in a test-tube add potassium or sodium-hydrate solution or slaked lime; shake well or warm it; the characteristic odor of Ammonia is evolved.

(3) To a few drops of a solution of an ammonium salt add a few drops of alcohol, a drop or two of hydrochloric acid (Reagent 5), and a like small quantity of solution of platinic chlorid; a yellow crystalline precipitate of the double chlorid of platinum and ammonium ( $\text{PtCl}_4, 2\text{NH}_4\text{Cl}$ ) will be produced.

(4) To a moderately strong solution of an ammonium salt add a strong solution of tartaric acid and shake or stir the mixture well; a white granular precipitate (acid tartrate of ammonium) will be formed.

(5) Evaporate a few drops of a solution of an ammonium salt, or place a fragment of an ammonium salt on a piece of porcelain and heat it; it is readily volatilized.

(6) Add Nessler's reagent in excess to a solution supposed to be ammoniacal; a brown precipitate or yellow color indicates the presence of Ammonia. This reaction is extremely delicate, and the estimation of Ammonia in drinking-water is founded upon it.

*Nessler's reagent* is made by dissolving 30 or 40 gms. (463 or 617 grs.) of potassium iodid in a small quantity of hot distilled water, adding a strong hot solution of mercuric chlorid (corrosive sublimate) until the pre-

precipitate of red mercuric iodid ceases to redissolve, even by the aid of rapid stirring and heat, slightly diluting, filtering, adding a strong solution of 120 to 140 gms. ( $3\frac{1}{2}$  to 4 oz.) caustic soda, or 160 to 180 gms. (5 to  $5\frac{1}{2}$  oz.) caustic potash, and diluting to one liter (34 f. oz.). A few c.c. of strong solution of mercuric chlorid (corrosive sublimate) are finally stirred in, the whole set aside till all precipitated red mercuric iodid has deposited, then decant the clear liquid for use.

(7) Ammonia is usually present in water as carbonate, but frequently in such small quantities that it cannot be detected by the ordinary tests. By mixing 2 to 3 liters of the water with 20 drops hydrochloric acid (Reagent 5), evaporating to dryness, dissolving the residue in 10 or 15 c.c. (162 or 243 minims) distilled water, filtering, and applying Bohlig's test, which consists in adding, first, 5 drops of solution of mercuric chlorid (corrosive sublimate), 1 part in 30 parts of water, and then 5 drops of solution of potassium carbonate, 1 part in 50 parts of water, when a cloudiness indicates the presence of Ammonia.

(8) Amidol (diamidophenol) gives a yellow color with solution of Ammonia of an intensity superior to that produced by Nessler's reagent, the color being easily seen in solutions containing 1 part of Ammonia in 1,000,000 parts of water.

(9) The evolution of even slight traces of Ammonia can be detected by the appearance of a dark-blue color on strips of filter-paper soaked in a 7 per cent solution of copper sulfate ( $\text{CuSO}_4$ ) and placed in the open end of the condenser at the beginning of distillation.

(10) To a solution containing Ammonia or its compounds (the latter previously made alkaline) add Einbrodt's reagent, consisting of a solution of mercuric chlorid (corrosive sublimate) to which a minute quantity of an alkaline carbonate has been added; a white precipi-

tate or cloudiness will be produced if the merest trace of Ammonia is present.

(11) Dissolve 0.010 gm. ( $\frac{1}{10}$  gr.) of tannic acid in 15 c.c. (4 f. drachms) of alcohol, and add a few drops of the solution to the liquid to be tested; a yellow or light-brown color indicates Ammonia.

### Test for Amygdalin, $C_{20}H_{27}NO_{11} + 3H_2O$ .

Amygdalin is a white crystalline powder of a slightly bitter taste; soluble in 12 parts of water and 150 parts of alcohol at the normal temperature; easily soluble in boiling alcohol and in hot water; nearly insoluble in ether, chloroform, and carbon disulfid. A watery solution is neutral to litmus paper.

(1) Warm a few grains of Amygdalin in a test-tube; it gives off water and melts to a brown liquid, which solidifies to a clear, cracked mass.

(2) Heat the above (1) mass; it chars and produces brown-colored vapors of an aromatic odor and acid reaction to moist litmus paper. Water shaken with the charred Amygdalin has also an acid reaction.

(3) Dissolve 0.005 gm. ( $\frac{1}{200}$  gr.) Amygdalin in 5c.c. (81 minims) of sulfuric acid (Reagent 21); a violet, red-colored, clear fluid is produced.

(4) Warm the (3) solution; it chars, and an odor of bitter-almond oil is developed.

(5) Evaporate a few drops of the (3) solution with a few drops of hydrochloric acid (Reagent 5); it gives a dark-brown residue; with nitric acid (Reagent 15) it leaves a colorless residue.

(6) Add a small quantity of Amygdalin to an alkaline cupric-tartrate solution (Fehling's solution) and warm it; cuprous oxid is thrown down and ammonia developed.

Prepare the alkaline cupric-tartrate solution as follows:

*Solution A.*—Crystals of pure copper sulfate powdered and pressed between filter-paper; 34.64 gm. (534 grs.) are weighed and dissolved in water; 0.5 c.c. (8 minims) of sulfuric acid (Reagent 21) added, and the solution diluted to 500 c.c. (17 f. oz.).

*Solution B.*—Dissolve 175 gm. (6 oz., 76 grs.) of Rochelle salt (potassium-sodium tartrate) in 350 c.c. (11 f. oz., 400 minims) of water; filter; add to the filtrate a clear solution of 50 gm. (1 oz., 334 grs.) of caustic soda in 100 c.c. (3 f. oz., 183 minims) of water. Dilute the solution to 500 c.c. (17 f. oz.). Preserve the solutions A and B separately and mix 1 c.c. (16 minims) of each for reaction when needed.

(7) Warm 0.10 gm. ( $1\frac{1}{2}$  grs.) of Amygdalin with 0.50 gm. (8 grs.) manganese dioxid, 2 c.c. (32 minims) sulfuric acid (Reagent 21), and 1 c.c. (16 minims) of water; the pungent odor of formic acid is developed; the fumes of the odor have an acid reaction to moist litmus paper. The mixture has an aromatic odor while cooling.

(8) Make an emulsion by rubbing two sweet almonds with 30 c.c. (1 f. oz.) of water, and dissolve 0.050 gm. ( $\frac{4}{8}$  gr.) of Amygdalin in it; the odor of bitter-almond oil and of hydrocyanic acid is noticed.

(9) Take a few lumps of granulated zinc, pour water with 20 per cent of sulfuric acid on it, and when hydrogen develops, quietly, dissolve 0.10 gm. ( $1\frac{1}{2}$  grs.) of Amygdalin in the fluid; a strong aromatic odor is developed, due to benzaldehyde, disappearing when the previously decanted fluid is warmed with a drop of nitric acid.

### Test for Amyl Nitrite, $C_5H_{11}NO_2$ .

Amyl Nitrite is produced by the action of nitric or nitrous acid on amylic alcohol, or by distilling together potassium nitrite, amylic alcohol, and sulfuric acid. It is

a clear, yellowish liquid of a peculiar, ethereal, fruity, suffocative odor; a pungent aromatic taste; specific gravity 0.865 to 0.875 at 25° C.; boils at about 96° to 99° C., yielding an orange-colored vapor, which explodes if heated above its boiling-point. It is almost insoluble in water, on which it floats; miscible in all proportions with alcohol or ether. In alcoholic solution it gradually decomposes, with formation of ethyl nitrite and amylic alcohol. It is very volatile, even at a low temperature, and is inflammable, burning with a fawn-colored, luminous, and sooty flame. It is neutral or slightly acid to litmus paper.

(1) By heating Amyl Nitrite with potassium hydrate it forms amylic alcohol and potassium valerianate.

(2) When Amyl Nitrite is shaken up with a solution of potassium iodid, acidulated with diluted sulfuric acid, iodine is liberated and nitric oxide (NO) is evolved.

(3) If 1 c.c. (16 minims) of normal potassium hydroxid T.S. and 10 c.c. (162 minims) of water be mixed in a test-tube with a drop of phenolphthalein T.S., then 5 c.c. (81 minims) of Amyl Nitrite be added, and the tube inverted a few times, the red tint of the aqueous layer should still be perceptible (limit of *free acid*).

(4) A mixture of 1.5 c.c. (25 minims) of silver nitrate T.S. and 1.5 c.c. (25 minims) of alcohol with a few drops of ammonia-water (Reagent 1) should not become brown or black if 1 c.c. (16 minims) of Amyl Nitrite be added and the mixture gently heated (absence of *aldehyde*).

(5) Amyl Nitrite should remain transparent or nearly so when exposed to the temperature of melting ice (absence of *water*),

### Test for Andromedotoxin, $C_{31}H_{51}O_{10}$ .

#### *Asebotoxin.*

Andromedotoxin is a poisonous glucoside obtained from some plants belonging to the Heath family, as *Andromeda japonica*, *Andromeda polifolia*, *Rhododendron ponticum*, etc. It is in colorless needles; soluble in water, alcohol, and in commercial amyl alcohol; very little in chloroform, much less in ether, and nearly insoluble in benzene. The watery solution has no effect on litmus paper. Pure Andromedotoxin gives off no odor during evaporation, but if not completely purified, a strong and very characteristic odor of ericinol is evolved.

(1) Warm a solution of Andromedotoxin in dilute sulfuric acid (1:5): it turns dark red; evaporate the solution: it gives a beautiful rose-red color.

(2) Warm Andromedotoxin with diluted hydrochloric acid: yellow, green, and violet colors are seen; evaporation of the solution gives a residue of a violet-red tint.

(3) Warmed with diluted phosphoric acid Andromedotoxin gives a red-brown color, and when evaporated with it gives a mulberry-red residue.

(4) Warm Andromedotoxin with diluted nitric acid, evaporate to dryness and expose to vapors of ammonia; a turmeric yellow color is seen.

### Test for Anilin, $C_6H_5NH_2$ .

#### *Amidobenzene. Phenylamine.*

Anilin was originally obtained from indigo, hence the name from the Portuguese word *anil*, indigo; but it is now largely manufactured from coal-tar or nitrobenzene. It is a thin, colorless, oily liquid, turning yellow or brown on exposure to the air; of a peculiar, vinous odor, and

sharp, aromatic taste; inflammable; sparingly soluble in water; soluble in all proportions in ether, alcohol, chloroform, and the oils, fixed and volatile; not in all proportions in carbon disulfid; neutral to red litmus paper, but faintly alkaline to violet dahlia paper, turning it green.

(1) Agitate strongly 2 c.c. (32 minims) of Anilin with 50 c.c. (1 f. oz., 324 minims) of water; filter. This solution gives a precipitate in 5 per cent solutions of ferric and ferrous salts, in 5 per cent zinc, and in 5 per cent alum salts.

(2) A trace of Anilin stirred with a glass rod the size of a match into 2 c.c. (32 minims) of sulfuric acid containing chromic acid (Reagent 22) colors the latter permanently blue. A watery solution of Anilin gives the same reaction, in course of time in the cold, quicker by warming. Neither chloroform, water, nor carbon disulfid dissolves the blue color.

(3) Potassium dichromate added in excess to Anilin solutions, acidulated with sulfuric acid (Reagent 21), causes a dark-green precipitate, turning black by further addition of the reagent.

(4) Equal volumes of Reagent 11 and the watery Anilin solution yields a clear mixture, in which crystals soon form.

(5) A proper quantity of a watery Anilin solution added to about 5 c.c. (81 minims) of mercuric-chlorid solution, 5% (Reagent 13), gives a crystalline precipitate.

(6) Three c.c. (48 minims) of mercuric potassium iodid solution (Reagent 12) and 3 c.c. (48 minims) of the watery solution of Anilin obtained under (1) cause no precipitate at first; very soon, however, crystalline needles form in the fluid; an amorphous precipitate is produced immediately if an excess of the Anilin solution is used, which precipitate is soluble by adding still more of the reagent.

(7) The yellow fluid obtained by adding 0.001 gm. ( $\frac{1}{64}$  gr.) of potassium chlorate to five drops of sulfuric acid (Reagent 21) becomes blue violet when a few drops of a watery solution of Anilin is added; the color fades slowly to red by diluting the mixture with water; ammonia-water (Reagent 1) restores the blue color.

(8) Chlorin-water (Reagent 3) added to watery Anilin solutions gives violet-colored fluids.

(9) Bromin-water (Reagent 2) added to watery Anilin solutions gives white, blue, red, or dirty-green colored fluids.

(10) Moisten a glass rod with sodium-hydrate solution (Reagent 20) and hold this for a few minutes in the vapor of chlorin in the chlorin-water bottle; a trace of sodium hypochlorid is thereby formed. A watery Anilin solution stirred with this rod becomes of a dirty-violet color; if the glass rod moistened with sodium-hydrate solution has been exposed to bromin vapor instead, the colors will be red-yellow, white when iodine-water is taken.

(11) A glass rod moistened with hydrochloric acid (Reagent 5) gives off white clouds when held in a test-tube containing a drop of Anilin. Stirring the Anilin with it, it forms a crystalline mass of Anilin hydrochlorate; when this is spread out on a mirror and a trace of potassium chlorate added, red, blue, and green colors are obtained.

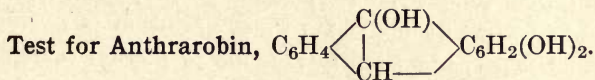
(12) Anilin gives the isonitril reaction described under *Acetanilid* (1) by taking Anilin instead of acetanilid.

(13) Add a solution of chlorinated lime (bleaching-powder) to the watery Anilin solution; a deep-blue or violet color is obtained.

(14) Acidulate the watery Anilin solution with one or two drops of sulfuric acid (Reagent 21) and add it to paper pulp made from wood fibre; an intensely yellow color is obtained.

(15) Make the watery Anilin solution faintly acid with sulfuric acid (Reagent 21), and put one or two drops of it on a piece of clean platinum foil, then touch it with the negative pole of a galvanic battery; the solution turns blue, then violet, and finally pink.

(16) To 1 c.c. (16 minims) of sulfuric acid (Reagent 21) add three or four drops of the watery Anilin solution (1); the mixture is first colored red and then blue by 0.065 gm. (1 gr.) of potassium dichromate.



Anthrarobin is produced from alizarin, a constituent of madder. It is a brownish-gray amorphous powder; soluble in ether, and in alcohol, with a yellowish-brown color; less soluble in chloroform; insoluble in carbon disulfid. Water dissolves very little, even at the boiling-point. It has an acid reaction to litmus paper.

(1) Add ferric-chlorid solution (Reagent 4) to the clear liquid to be tested: a dirty green-brown color indicates Anthrarobin.

(2) Add lime-water to a solution of Anthrarobin; it turns a reddish brown.

(3) Drop some of the clear solution of Anthrarobin on Reagent 21; it turns violet-red.

(4) Drop some of the solution (3) on Reagent 22; it gives a brown-red color.

(5) Add a few drops of the watery solution of Anthrarobin to a little nitric acid (Reagent 15); it is decomposed and turns red with a dark-brown mass.

(6) Lead-acetate and lead-subacetate solutions give in the watery solution of Anthrarobin brown precipitates.

(7) Reagent 17 added to the watery solution gives a strong dark-brown turbidity.

(8) 0.005 gm. ( $\frac{1}{2}$  gr.) Anthrarobin, 2 c.c. (32 minims) water, and 5 drops of ammonia-water (Reagent 1) give a greenish-brown color, changing to blue and violet.

**Test for Antipyrin—Phenyldimethylpyrazolone—Phenazone—Analgesin,  $C_3H(CH_3)_2N_2(C_6H_5)O$ .**

Antipyrin is a colorless crystalline substance with a bitter taste, freely soluble in water, alcohol, and chloroform; less soluble in ether; insoluble in petroleum spirit and in carbon disulfid. Its watery solution is neutral to litmus paper, but slightly alkaline to methyl orange.

(1) Tannin (Reagent 24) causes a strong precipitate in a 1 per cent solution of Antipyrin.

(2) To 10 c.c. (162 minims) of a watery solution of Antipyrin add a few drops of a saturated solution of picric acid (Reagent 16); yellow prismatic crystals commence to form very soon if it contains only  $\frac{1}{2}$  per cent of Antipyrin. In a stronger solution the picric acid causes an immediate precipitate.

(3) Add 5 c.c. (81 minims) of mercuric potassium iodid solution (Reagent 12) to 10 c.c. (162 minims) of a 2 per cent watery solution of Antipyrin; this gives a turbidity; if it contains 5 per cent feathery crystals will form in the course of half a day.

(4) 1 c.c. (16 minims) of a 5 per cent mercuric-chlorid solution (Reagent 13) added to 4 c.c. (64 minims) of a 2 per cent Antipyrin solution gives an amorphous precipitate, disappearing on warming slightly, reappearing when the fluid cools off.

(5) To 4 c.c. (64 minims) of concentrated sulfuric acid (Reagent 21) add an equal volume of the watery solution of Antipyrin and warm it; the acid remains colorless; now add a small crystal of sodium nitrate ( $NaNO_3$ ); a permanent yellow or reddish color is produced.

(6) Antipyrin triturated with sugar turns yellow when a few drops of sulfuric acid are added to the mixture in the mortar.

(7) 2 c.c. (32 minims) of nitric acid (Reagent 15) dissolve 0.20 gm. (3 grs.) Antipyrin at 15° C. without coloring; the slightest heat colors the fluid yellow, turning into a permanent red; this red solution, when spread out in a thin film, may be evaporated to dryness without losing its color.

(8) A solution of 0.30 gm. (5 grs.) Antipyrin and 0.10 gm. (1½ grs.) sodium nitrite ( $\text{NaNO}_2$ ) in 2.5 c.c. (40 minims) of water turns greenish blue on the addition of 10 drops of acetic acid (1.064 sp. gr.); in the course of an hour green crystals of nitrosoantipyrin appear in the fluid; the crystals are bluish green by lamplight.

(9) The same reaction (8) occurs when Antipyrin is added to sweet spirit of nitre (ethyl nitrite) containing free acid.

(10) 0.10 gm. (1½ grs.) Antipyrin, 0.10 gm. (1½ grs.) sodium nitrite ( $\text{NaNO}_2$ ), and 0.20 gm. (3 grs.) mercuric chlorid ( $\text{HgCl}_2$ ), shaken together with 4 c.c. (65 minims) water, turn green (nitrosoantipyrin) in the course of an hour.

(11) Add one drop of a saturated watery solution of Antipyrin to 1 c.c. (16 minims) of Reagent 4; this produces a flocculent precipitate soluble in water or alcohol to a blood-red fluid; sodium chlorid ( $\text{NaCl}$ ) is conducive to the formation of the flocculent precipitate.

(12) A red or yellowish-red amorphous precipitate is formed on mixing 100 c.c. (3 f. oz.) of a saturated watery solution of sodium chlorid ( $\text{NaCl}$ ) with 1 c.c. (16 minims) of a saturated watery solution of Antipyrin and 1 c.c. (16 minims) of Reagent 4.

(13) To 5 c.c. (81 minims) of a 10 per cent watery solution of Antipyrin, add chlorin-water (Reagent 3); this

causes a turbidity, immediately disappearing and especially noticeable along the sides of the test-tube.

(14) Add two drops nitric acid (Reagent 15) to 2 c.c. (32 minims) of a solution of Antipyrin; it turns green, changing to red by adding three drops more and boiling.

(15) Heat a little Antipyrin with zinc chlorid until vapors are given off; an odor like that of methylamine or cacodyl, and a reddish-yellow residue with greenish fluorescence by reflected light are obtained.

(16) Antipyrin shows two characteristic color reactions; with ferric-chlorid solution (Reagent 4) it is colored deep red, which color is changed by sulfuric acid (Reagent 21) into bright yellow; with nitrous acid ( $\text{HNO}_2$ ) a green color, and in concentrated solutions a separation of green crystals of nitrosoantipyrin. Both these reactions are adapted for the recognition of it in urine.

### Test for Apocodein, $\text{C}_{18}\text{H}_{19}\text{NO}_2$ .

Apocodein is a brown, amorphous powder; soluble in ether, alcohol, and chloroform; insoluble in carbon disulfid and in cold water; slightly soluble in boiling water; gives an alkaline reaction to moist red litmus paper.

(1) Ferric-chlorid solution (Reagent 4) gives a greenish-brown precipitate in an alcoholic solution of Apocodein that is soluble in alcohol and in water.

(2) Reagent 4 added to a solution of Apocodein, made with boiling water, gives a brown fluid, turning green.

(3) Chlorin-water (Reagent 3) dissolves Apocodein with a yellow color, turning red after adding ammonia-water (Reagent 1).

(4) The following color reactions are made with acids: red with sulfuric (Reagent 21); red with nitric (Reagent 15); dark brown with sulfuric containing chromic acid (Reagent 22).

(5) Acetic acid added to a mixture of Apocodein with peroxid of manganese and water turns the latter green; filter; agitate with chloroform; the color of the filtrate changes to blue, and a brown-colored top layer is formed. Substituting peroxid of lead for the peroxid of manganese, we obtain a red-brown filtrate, which chloroform does not change.

(6) Triturate 0.020 gm. ( $\frac{5}{16}$  gr.) ammonium molybdate with five drops sulfuric acid (Reagent 21), and add to it a very small quantity of Apocodein; the color is brownish black at first and changes slowly to blue.

(7) Dissolve 0.065 gm. (1 gr.) of potassium ferricyanid in 200 c.c. (6 f. oz., 360 minims) of water, to which 1 c.c. (16 minims) of ferric-chlorid solution (Reagent 4) is added; a few particles of Apocodein moistened with the mixture give immediately a blue precipitate.

(8) Apocodein gives a mirror with  $\frac{N}{10}$  silver volumetric solution; the test to be made in the dark.

(9) Dissolve 0.10 gm. ( $1\frac{1}{2}$  grs.) potassium iodate ( $KIO_3$ ) in 5 c.c. (81 minims) of water; add 0.005 gm. ( $\frac{1}{4}$  gr.) of Apocodein, and finally 5 drops of acetic acid (1.06 sp. gr.); this produces a yellowish fluid, changing to green; agitate a part with carbon disulfid—this becomes amethyst-colored; another part with chloroform—this colors blue.

### Test for Apomorphin Hydrochlorate, $C_{17}H_{17}NO_2 \cdot HCl$ .

Apomorphin Hydrochlorate, as usually seen, is a gray or greenish-gray powder; it should be in white, shining crystal needles. It is odorless; has a slightly bitter taste; soluble in 30 parts water at normal temperature; in 40 parts of alcohol (0.83 sp. gr.); very sparingly in ether or chloroform. The watery solution is neutral and colorless, but turns green on exposure to air and light. Ether re-

moves the alkaloid from the green-colored watery solution, with a purple-red color; chloroform with a beautiful blue hue.

(1) Reagents 6, 12, 13, and 14, also solutions of sodium chlorid, potassium bromid, and potassium iodid form precipitates, which turn green, when added to a watery solution of Apomorphin Hydrochlorate.

(2) Ammonia-water (Reagent 1) dissolves Apomorphin Hydrochlorate with a brown color which turns reddish.

(3) Lime-water dissolves it with a green color, afterwards changing into red and brown; very soon a greenish-brown sediment settles down.

(4) Apomorphin Hydrochlorate is soluble in iodine-water (Reagent 6) with a red color, changing to green.

(5) The filtrate of the colorless watery solution of Apomorphin Hydrochlorate turns green when shaken with peroxid of manganese; red when peroxid of lead is taken; and green, changing to a pink-red and brown color, when peroxid of barium is taken. Carbon disulfid, ether, or benzene extract a constant purple or red color from all these green fluids; with chloroform, a splendid blue solution is obtained.

(6) Dissolve 0.005 gm. ( $\frac{1}{200}$  gr.) Apomorphin Hydrochlorate in 2 c.c. (32 minims) acetic acid; the solution is colorless, but turns blood-red on shaking with a small crystal of potassium iodate (Reagent 9); ether and chloroform extract an indigo-blue color, and carbon disulfid extracts iodine with its usual amethyst color.

(7) A blue fluid is obtained in the course of an hour by agitating 0.050 gm. ( $\frac{1}{20}$  gr.) Apomorphin Hydrochlorate, 0.050 gm. ( $\frac{1}{20}$  gr.) ferrous sulfate, and 10 c.c. (162 minims) of water; the next day the fluid is blue-black, changing to dirty green.

(8) 0.001 gm. ( $\frac{1}{1000}$  gr.) Apomorphin Hydrochlorate produces a mirror from reduced silver in 0.5 c.c. (8 minims)

of a  $\frac{N}{10}$  silver volumetric solution in the course of half a day; more quickly when ammonia-water (Reagent 1) is added.

(9) Dissolve 0.065 gm. (1 gr.) potassium ferricyanid in 200 c.c. (6 f. oz., 360 minims) water; add 1 c.c. (16 minims) ferric-chlorid solution (Reagent 4); a drop or two of this mixture added to a few crystals of Apomorphin Hydrochlorate on a porcelain lid of a crucible gives a precipitate of Prussian blue immediately.

### Test for Arbutin, $(C_{12}H_{16}O_7)_2 + H_2O$ .

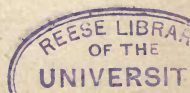
Arbutin crystallizes in long colorless needles, united in tufts; soluble in 8 parts of water at normal temperature; in much less at  $100^\circ C$ .; soluble in 16 parts alcohol (0.83 sp. gr.); nearly insoluble in ether, chloroform, and carbon disulfid. The solutions have a bitter taste and a neutral reaction to litmus paper. It is a glucoside from uva ursi.

(1) Arbutin gives color reactions with acids: yellow changing to red-brown with sulfuric acid (Reagent 21); yellow with nitric acid (Reagent 15).

(2) A mixture of 0.050 gm. ( $\frac{4}{5}$  gr.) Arbutin and 1 gm. ( $15\frac{1}{2}$  grs.) peroxid of manganese heated in a glass tube gives off acid vapors to moist blue litmus paper, and likewise an odor of quinon.

(3) Dilute on a porcelain lid a drop of ferric-chlorid solution (Reagent 4) with water until the peculiar yellow color of the reagent can hardly be noticed, when the addition of Arbutin produces a light-blue color.

(4) When phosphomolybdic acid is added to a solution of Arbutin previously rendered alkaline by ammonia-water (Reagent 1) or other alkali, a blue color is produced, which is deep when the solutions are strong, but observable even when they are very dilute.



**Test for Aristol—Di-iododithymol,  $C_{20}H_{24}I_2O_2$ .**

Aristol is a flesh-colored or reddish-brown amorphous powder, prepared by decomposing a solution of iodine in potassium iodide by an alcoholic solution of thymol; it is nearly odorless and tasteless; insoluble in water, sodium hydrate solution (Reagent 20), and in glycerin, even at  $100^{\circ}C$ .; slightly soluble in alcohol and ether; easily soluble in chloroform, carbon disulfide, and olive-oil, with a yellow-red color; does not affect litmus paper.

(1) Strewn on sulfuric or nitric acid, Aristol does not seem to change. A red solution is obtained when sodium nitrate ( $NaNO_3$ ) is added to the sulfuric acid, and a blue solution when sodium nitrite ( $NaNO_2$ ) is added.

(2) Ferric-chloride solution (Reagent 4) has no effect on an alcoholic solution of Aristol; when the solution is evaporated to dryness on a water-bath, however, it leaves a black residue that is soluble with dark-brown color in chloroform and in carbon disulfide, but insoluble in water.

(3) Carbon disulfide does not extract iodine from a filtrate of Aristol when the latter has been strongly shaken with cold water, not even after the addition of bromine-water (Reagent 2); after the water is warmed, shaken again with Aristol, and filtered, the result is colorless as before, but now carbon sulfide and bromine-water show the presence of iodine in the cold filtrate.

(4) The presence of iodine can also be proved by a piece of moistened writing-paper suspended in the vapor of hot water that is shaken with Aristol.

**Test for Arsenic,  $As_2O_3$ .**

Arsenic is a white, tasteless, odorless powder; entirely volatilized by heat; sparingly soluble in cold water, alcohol, and oils; more soluble in boiling water and gly-

cerin; freely soluble in hydrochloric acid and in solutions of alkalies and carbonates. Its aqueous solution feebly reddens blue litmus paper. The presence of organic matters very much impairs the solvent power of water for Arsenic, a circumstance which readily explains why it has not, in some cases, been found in the liquid contents of the stomach of persons poisoned by it.

(1) Introduce into a reduction-tube any solid compound of Arsenic, including Paris green, the two sulfids, and any arsenite, and cover it with six times the quantity of a well-dried mixture of 3 parts of sodium carbonate to 1 part of potassium cyanide; heat gently. Some moisture may first appear on the tube; this can be removed with a spiral of filter-paper, a swab of absorbent cotton, or by gently heating the moist glass. When the tube is dry, strong heat is applied to the flux and then to the compound of Arsenic. The compound is reduced to metallic Arsenic, with an odor of garlic, and is deposited higher up on the tube as a mirror-like ring, black, shading to brown or gray. Charcoal, or a mixture of charcoal and potassium carbonate, may be substituted for the sodium carbonate and potassium cyanide.

(2) Hold the flame under the mirror-like ring (1); it disappears and forms a deposit of white octahedral crystals on a cooler part of the tube highly characteristic of Arsenic.

(3) Boil the octahedral crystals (2) in a little water; add to it a few drops of ammonio-nitrate of silver solution; a canary-yellow precipitate of silver arsenite ( $\text{Ag}_3\text{AsO}_3$ ) is deposited which dissolves in ammonia-water (Reagent 1) and in nitric acid (Reagent 15), but not in sodium hydrate solution (Reagent 20). This is Hume's test.

*Ammonio-nitrate of silver solution* is prepared by freshly diluting ammonia-water and adding to it a strong solu-

tion of silver nitrate until the precipitate of silver oxide formed ceases to dissolve.

(4) Put 1 c.c. (16 minims) ammonia-water (Reagent 1) in a test-tube and dilute it with 10 c.c. (162 minims) water. To this dilute ammonia-water add a weak solution of copper sulfate until the bluish-white precipitate ceases to dissolve. The clear blue solution added to a solution of arsenic trioxid will throw down a bright-green precipitate of cupric arsenite ( $\text{CuHAsO}_3$ ), Scheele's green.

(5) A freshly made solution of stannous chlorid is added to the suspected material dissolved in strong hydrochloric acid (Reagent 5). Having immersed a small piece of pure tin-foil, the mixture is heated; if Arsenic be present, a brown color or a gray-brown precipitate of the metal is formed. This is Bettendorff's test.

(6) Arsenic sprinkled on red-hot charcoal burns with a blue flame and garlic odor.

(7) Generate hydrogen sulfid in the usual way by acting on iron sulfid with diluted sulfuric acid. Filter the liquid to be tested, and acidulate it with hydrochloric acid (Reagent 5); then pass the hydrogen-sulfid gas through it; after the gas has passed through the liquid for a few minutes, yellow arsenious sulfid ( $\text{As}_2\text{S}_3$ ) is precipitated if it contains Arsenic. The separation of the precipitate is promoted by boiling and exposure of the liquid for a few hours to the air; the precipitate is soluble in ammonium sulfid, but insoluble in strong boiling hydrochloric acid; dried and heated in a small test-tube with a mixture of sodium carbonate and potassium cyanide, it yields a mirror of metallic Arsenic.

(8) In a test-tube containing 1 c.c. (16 minims) of the suspected solution, either acid or neutral, put about 1 gm. ( $15\frac{1}{2}$  grs.) of chemically pure zinc and 5 c.c. (81 minims) of a 6 per cent dilution of sulfuric acid. In the upper

part of the test-tube insert a plug of absorbent cotton moistened with lead-acetate solution, and clasp over the mouth of the test-tube a cap made of three layers of filter-paper. Having wet only the upper layer with a drop of saturated solution of silver nitrate, set aside in a dark box for a time. Arsenic will cause on the paper a bright-yellow spot, which darkens, by separation of metallic silver, when water is applied to it. This is Gutzeit's test.

(9) A piece of bright copper-foil should be put into pure water containing one-sixth part of hydrochloric acid (Reagent 5), and then heat applied so as to boil for five minutes. The copper remaining bright, the hydrochloric acid may be assumed to be pure. Every detail of this test and others must be paralleled by blank experiments before the analyst can be sure. Having added one-sixth volume of hydrochloric acid to the solution to be tested, the pure copper-foil is put into it and the whole boiled for a few minutes. If Arsenic be present, it is deposited as a dark film, purple to steel-gray in color. Reinsch's test.

(10) Generate hydrogen in the usual manner, from water by zinc, or magnesium, and sulfuric acid (Reagent 21) with one or two drops of platinic-chlorid solution; allow the gas to escape through a small tube; when all the air has been expelled (which should be determined by collecting a small test-tube full and holding its mouth to a flame; if the gas burns quietly without explosion it is pure), ignite the escaping gas and hold a piece of earthenware in the flame; if no brown spot is deposited on the earthenware, pour eight or ten drops of the suspected liquid into the funnel-tube, washing the liquid into the generating-bottle with a little water; if organic matter should cause much frothing, a small quantity of alcohol may be introduced by the funnel-tube. Imme-

diately hold the earthenware again in the flame; if a brown spot is deposited, add to it a drop of solution of chloride of lime (bleaching-powder); it quickly dissolves if it is Arsenic. This is Marsh's test.

(11) Into a large test-tube place a piece of pure zinc weighing about 1 to 1.25 gm. (15½ to 19 grs.); add about 10 c.c. (162 minims) of strong solution of caustic soda or potash; cover the mouth of the test-tube with filter-paper moistened with a drop of solution of silver nitrate and set it aside for two hours in a dark place. Now, if the solution of silver nitrate has not darkened, add a few drops of the liquid to be tested, and again set it aside in the dark; a black or brown spot reveals the presence of Arsenic. Aluminium or magnesium may be used instead of the zinc. This is Fleitmann's test.

(12) Arsenic added to distilled water in a test-tube and boiled dissolves very slowly; part of it floats on the surface of the water or aggregates in small lumps at the bottom.

(13) To identify the presence of Arsenic in wall-paper dissolve the coloring-matter off in a little ammonia-water, pour off this solution on a piece of glass and drop into the liquid a crystal of silver nitrate; a yellow coloration around the crystal indicates the presence of Arsenic.

(14) If organic substances (as the stomach, liver, spleen, food, etc.) are to be examined for the poison, cut them into small fragments, bruise these in a mortar, and boil with distilled water acidulated with about one-tenth of pure hydrochloric acid (Reagent 5), adding from time to time small portions of potassium chlorate until the solids are all dissolved or broken down into fine flakes or grains, and continue a gentle heat until the odor of chlorin disappears. Then make a dialyzer by folding a circular sheet of parchment-paper like a plain filter, stand it in a bowl on its point, supported by a frame, and pour half of the

solution into it; then pour enough distilled water into the bowl, outside of the dialyzer, to be on a level with the solution, and set aside for forty-eight hours. Then carefully evaporate the dialyzate to one-fourth its volume and test it with some of the reagents above described. Strain the other half of the solution through muslin, heat to the boiling-point, and subject it to Reinsch's test. Nitric acid (Reagent 15), assisted by sulfuric acid (Reagent 21), is sometimes used to destroy the organic matter and oxidize the Arsenic into arsenic acid. In such a case the arsenic acid can be readily extracted by boiling water, and the solution filtered, evaporated to dryness, and tested with some of the above reagents.

(15) Half fill a test-tube with the liquid to be tested, add a few drops of liquor calcis (lime-water); a white precipitate (arsenite of lime), soluble in acids, indicates the presence of Arsenic.

(16) Dissolve 25 gm. (386 grs.) of stannous chlorid in a mixture of 100 c.c. (3 f. oz., 183 minims) of ether and 20 c.c. (324 minims) of hydrochloric acid, and decant the clear solution; add 5 c.c. (81 minims) of this to an equal volume of the liquid to be tested, and heat for one minute at 40° C.; a brownish-red ring at the point of contact of the two liquids indicates Arsenic.

(17) To detect Arsenic in glycerin dissolve 20 gm. (308 grs.) of sodium hypophosphite in 20 c.c. (324 minims) of water, add 200 c.c. (6 f. oz.) of concentrated hydrochloric acid (sp. gr. 1.17), filter through cotton, and mix 10 c.c. (162 minims) of this reagent with 5 c.c. (81 minims) of the glycerin to be tested. On now heating the mixture on a water-bath, a flocculent brown precipitate or dark-brown color develops if but 0.0001 gm. ( $\frac{1}{10000}$  gr.) of arsenious acid is present.

### Test for Atropin, $C_{17}H_{23}NO_3$ .

Atropin crystallizes in white prismatic needles, gradually becoming yellowish on exposure to the air; odorless; having a bitter, acrid taste; soluble in 450 parts of cold water and 60 parts of boiling water; very soluble in alcohol, chloroform, ether, and glycerin; very little in carbon disulfid; placed on red litmus paper, moistened with alcohol, it has hardly an alkaline reaction, none at all on phenolphthalein paper; it is distinctly alkaline to both reagents, however, when touched with a drop of water.

(1) Mercuric oxid ( $HgO$ ) is thrown down when 0.001 gm. ( $\frac{1}{84}$  gr.) Atropin is dissolved in 1 c.c. (16 minims) alcohol (0.83 sp. gr.) and 1 c.c. (16 minims) of mercuric-chlorid solution (Reagent 13) is added, the whole being warmed on a watch-glass; when the Atropin is added afterwards, however, no such reaction takes place.

(2) Reagents 12 and 14 produce precipitates in a watery solution of Atropin, amorphous at first, but very soon taking on crystalline form. These forms are characteristic.

(3) Tannin (Reagent 24) produces, in watery solutions of Atropin, a white curdy precipitate that is soluble in excess of the reagent, in acetic acid, and in ammonia-water (Reagent 1), but not in excess of the Atropin solution.

(4) Picric acid (Reagent 16) causes a precipitate only in Atropin solutions made with boiling water.

(5) A solution of iodine in potassium iodid (Reagent 7) gives a precipitate in an acidulated watery solution of Atropin.

(6) By boiling 0.10 gm. ( $1\frac{1}{2}$  grs.) Atropin with 3 c.c. (48 minims) water and 0.20 gm. (3 grs.) calomel, the latter turns black; filter boiling-hot, and supersaturate the

alkaline fluid decanted from the crystals that form by cooling with nitric acid (Reagent 15); add silver nitrate solution, 5 per cent; this gives a voluminous precipitate.

(7) Heat 0.001 gm. ( $\frac{1}{64}$  gr.) Atropin in a small test-tube until white vapors arise; add 1 c.c. (16 minims) sulfuric acid (Reagent 21); heat a little longer, until the acid commences to color; add, along the sides of the test-tube, and at a safe distance from the operator, 2 c.c. (32 minims) water, drop by drop; note the odor, suggestive of flowers. Repeat this reaction, but drop a small crystal of potassium permanganate ( $\text{KMnO}_4$ ) into the acid solution before the water is added; in this case an odor of bitter almonds will be noticed. A trace of potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) produces yet another odor. An aromatic odor is also obtained from the decomposition products of Atropin and hydrochloric acid (Reagent 5) or phosphoric acid ( $\text{H}_3\text{PO}_4$ ) and even after boiling it repeatedly with water, evaporating, and redissolving the residue in hot water.

(8) Dissolve 0.001 gm. ( $\frac{1}{64}$  gr.) Atropin in a drop of nitric acid (Reagent 15); evaporate to dryness; prepare an alcoholic solution of potassium hydrate ( $\text{KOH}$ ) (1 gm. ( $15\frac{1}{2}$  grs.) : 5 c.c. (81 minims)); a drop of this solution added along the side of the porcelain dish or lid produces a violet color when it comes in contact with the residue; this test does not succeed in the presence of strychnin.

(9) Dissolve 0.010 gm. ( $\frac{1}{8}$  gr.) Atropin in very dilute hydrochloric acid; warm this and add a 5 per cent gold-chlorid solution; set aside to cool; Atropin gold chlorid solidifies to a granular mass.

### Test for Berberin, $(\text{C}_{20}\text{H}_{17}\text{NO}_4)_2 + 9\text{H}_2\text{O}$ .

Berberin is the active principle of *Barberry*; it is in small bright-yellow or red-yellow (orange-colored) needles; soluble in water, 1:300 parts, with a canary-yellow or orange

color, and in alcohol; insoluble in ether, chloroform, or carbon disulfid. These solutions do not affect litmus paper.

(1) Neither ammonia-water (Reagent 1), lime-water (Reagent 10), borax, or potash solutions have an effect upon paper colored yellow and dried with a solution of Berberin; turmeric paper turns red-brown when thus treated. Berberin solutions give a permanent yellow color to paper when absorbed and dried.

(2) Crystals of Berberin nitrate begin to form on adding 1 c.c. (16 minims) of nitric acid (Reagent 15) to 3 c.c. (48 minims) of a watery solution of Berberin; the color remains unaltered; becomes dark red by mixing equal volumes of Berberin solution with nitric acid, but no crystals are formed.

(3) Crystals of Berberin hydrochlorate or Berberin sulfate are formed when hydrochloric or sulfuric acid is added to a watery or alcoholic Berberin solution.

(4) Berberin and its salts give an olive-green color reaction when warmed with sulfuric acid (Reagent 21).

(5) A fragment of sodium nitrate, or of potassium dichromate (the first salt to be preferred, as it is colorless), added to a cold solution of Berberin in sulfuric acid (Reagent 21) causes violet streaks to flow in the acid when the mixture is stirred with a small glass rod.

(6) Bromin-water (Reagent 2) and chlorin-water (Reagent 3) give a purple zone when added to a watery solution of Berberin mixed with an equal volume of sulfuric acid (Reagent 21)

(7) 0.020 gm. ( $\frac{1}{3}$  gr.) of ammonium molybdate triturated with 5 drops of sulfuric acid (Reagent 21) gives a green color reaction, changing immediately to brown-green with Berberin.

(8) Iodin-water (Reagent 6) gives a greenish-red fluorescent precipitate in a solution containing Berberin.

(9) One drop of yellow ammonium polysulfid  $((\text{NH}_4)_2\text{S}(\text{NH}_4)\text{HS})$  gives a flocculent red-brown precipitate in 5 c.c. (81 minims) of a solution of 1:1000 of Berberin.

(10) 10 c.c. (162 minims) of a solution of 0.010 gm. ( $\frac{1}{8}$  gr.) Berberin in water are discolored in a few hours when slightly warmed with 10 c.c. (162 minims) of diluted sulfuric acid (sp. gr. 1.1) and 5 gm. (77 grs.) granulated zinc (zinc-dust is preferable) until hydrogen develops quietly and steadily; nitric acid restores a color reaction of red or yellow-red in this fluid.

### Test for Betol, $\text{C}_{10}\text{H}_7\text{O} \cdot \text{CO} \cdot \text{C}_6\text{H}_4 \cdot \text{OH}$ .

*Naphthalol: Naphthosalol:  $\beta$ -Naphthylsalicylate.*

Betol, a derivative of salicylic acid, occurs in white shining crystals; tasteless; having a faint, agreeable, aromatic odor slightly suggestive of salol; soluble in ether, chloroform, and carbon disulfid; in alcohol only when warm; also in fatty oils; in water with difficulty even at  $100^\circ \text{C}$ .; does not act upon litmus paper.

(1) Betol thrown upon sulfuric acid (Reagent 21) colors the latter yellow, subsequently changing slowly to brown.

(2) Betol gives no color reaction with nitric acid (Reagent 15) in the cold; at the boiling-point a flocculent sediment or red drops are formed.

(3) A mixture of 2 c.c. (32 minims) of sulfuric acid (Reagent 21), 0.20 gm. (3 grs.) of Betol, and 0.10 gm. ( $1\frac{1}{2}$  grs.) of chloral hydrate has a brown-red color.

(4) By adding sodium nitrate to the sulfuric-acid test (1), Betol produces a greenish-brown coloring; substituting sodium *nitrite* for the nitrate, the color is a more reddish brown; but by substituting nitric acid the sulfuric acid remains yellow.

(5) Betol added to Reagent 22 produces only a yellow color when warmed upon the water-bath.

(6) Betol is insoluble in sodium-hydrate solution (Reagent 20) even at the boiling-point.

(7) Betol warmed with a little slacked lime and water gives a blue fluorescent filtrate; this remains clear when slightly acidulated with hydrochloric acid (Reagent 5); one drop of ferric-chlorid solution (Reagent 4) added to it produces a violet color. One drop ferric-chlorid solution (Reagent 4) gives the same color to 5 c.c. (81 minims) of water that has been boiled for some time with a few crystals of Betol; replacing the water that evaporates, the same reagent colors an alcoholic solution of Betol brown, however. Betol triturated with Reagent 4 gives no coloring at all, not even by warming the mixture. •

(8) 0.010 gm. ( $\frac{1}{10}$  gr.) Betol gives a blue color reaction with 2 c.c. of sulfuric acid (Reagent 21) heated nearly to the boiling-point; a yellow-green clear fluid is obtained by adding water to the same.

(9) Ammonia-water (Reagent 1) does not give the fluorescent reaction described under (7) when agitated with Betol, but it becomes yellow by warming. An unctuous dark-brown sediment is obtained when the Betol is filtered off and the ammoniacal filtrate evaporated to about one-third; crystals of salicylic acid form in the cooling fluid.

### Test for Bile in Urine.

Urine containing Bile-pigment is always tinged of a deep-brown, reddish-yellow, greenish-brown, or dark- or grass-green color. Much froth is formed in it when shaken, and it imparts a yellow or greenish color to a slip of filtering-paper when dipped into it.

(1) Concentrated nitric acid slightly decomposed by exposure to the light is poured to about an inch high into a conical-shaped test-glass and a little of the urine to be tested carefully spread over its surface by means of a pipette, pouring it on the border of the glass. If cholepyrrhine is present the play of colors commences, at the line where the fluids come in contact, with a beautiful green ring, which gradually extends upwards, and at its under surface exhibits a blue, violet-red, and, lastly, a yellow ring. It should, however, be observed that the whole of these colors do not invariably appear; violet and green generally last the longest, but the green, which appears almost at the commencement of the action, is alone demonstrative of the presence of Bile-pigment. The presence of albumen in no way interferes with this test; a portion of the pigment is generally precipitated with the albumen, which is coagulated by the nitric acid, but it also beautifully shows the reaction. The nitric acid must not contain too much nitrous acid, for if it does the reaction is violent and the play of colors rapidly passes away. Gmelin's test.

(2) The slightest traces of Bile-pigment may be discovered by shaking large quantities of urine successively with chloroform and pouring off the exhaustive urine. The smallest quantity of cholepyrrhine present in the urine is taken up by the chloroform, which, when left at rest, by reason of its high specific gravity, rapidly sinks to the bottom, of a yellowish color. The supernatant urine is drawn off and a little nitric acid containing nitrous acid spread over the chloroform solution. If the slightest trace of cholepyrrhine be present, the reaction will then take place (from above downwards), and in a very brilliant form. The reaction with nitric acid in the chloroform solution is excessively delicate and beautiful.

(3) Gradually mix the urine with half its bulk of strong sulfuric acid (Reagent 21) in a test-tube, rise of temperature being prevented by dipping the test-tube in water. A small quantity of powdered white sugar is then introduced and well mixed with the acid urine, and more sulfuric acid (Reagent 21) then poured in; as the temperature rises, a reddish or violet coloration is produced. This is Pettenkofer's test.

(4) Put into a test-tube a mixture of 30 minims of nitric acid (Reagent 15) with 30 minims of the urine; pour into the test-tube 30 minims of strong sulfuric acid (Reagent 21); the presence of Bile-pigments is shown by the appearance of a grass-green ring at the plane of contact of the fluids. This is Fleicht's test.

(5) Evaporate 500 c.c. (17 f. oz.) of the urine nearly to dryness in a water-bath; extract the residue with alcohol, evaporate the alcohol, and dissolve the residue in a little water; treat the solution with lead acetate; collect the precipitate after standing twelve hours; wash it, and dry it between folds of filter-paper; extract the Bile salt of lead with boiling alcohol; add sodium carbonate, and evaporate to dryness; treat the residue with alcohol; add sulfuric acid (Reagent 21) to the soda solution, it becomes brownish red, then light blue or violet, and when heated with a little sugar, reddish or yellowish brown. Add two or three drops of sugar solution (1 part sugar to 4 parts water) to the soda solution, which is concentrated as much as possible, and then pure sulfuric acid (Reagent 21) free from sulfurous acid. If any Bile-acid is present, the fluid will first become muddy, then clear, and at the same time yellow, soon afterwards of a pale cherry, dark carmine-red, and lastly of a beautiful purple-violet color.

The reaction becomes much more sensitive when the soda solution is evaporated to a few drops in a porcelain

cup, a few drops of pure diluted sulfuric acid (1 part  $\text{H}_2\text{SO}_4$  to 4 parts  $\text{H}_2\text{O}$ ) and a trace of sugar solution added to it, and the mixture then carefully evaporated at a very gentle heat over a small lamp. We cannot be certain that Bile-acid is present unless the fluid assumes a distinct *purple-violet*, as well as a red color. This is Neakomm's test.

### Test for Blood in Urine.

Blood imparts to the urine the reaction of albumen and a red, brown, or smoky color.

(1) Add to the urine one-third its volume of sodium- or potassium-hydrate solution and boil it; the precipitated phosphates carry the Blood coloring-matter with them to the bottom in the form of red clouds. This is Heller's test.

(2) Prepare a tincture of guaiacum from the inner un-oxidized portions of guaiacum resin and an ethereal solution of hydrogen peroxide. Add to one or two drachms of the urine in a test-tube nearly as much as the ethereal solution and then two or three drops of the tincture of guaiacum; a bluish-green layer appears at the junction of the fluids if Blood is present.

(3) Acidulate the urine strongly with acetic acid and shake with an equal volume of ether. Should an emulsion form, the separation of the ether may be facilitated by cooling in ice-water, or by adding a few drops of alcohol. The ethereal extract is transferred to a test-tube in which a few drops of water have been dropped, and then 15 to 30 drops of old turpentine-oil or 5 to 10 drops of fresh hydrogen dioxide added, the whole lightly shaken, then 10 to 20 drops of an alcoholic 2 per cent solution of barbaloin added, and the mixture thoroughly shaken. The aqueous layer acquires a distinct red color in from one to three minutes if traces of Blood are present.

### Test for Bromoform, $\text{CHBr}_3$ .

Bromoform is produced by the action of bromin upon alcohol in the presence of an alkali; milk of lime is saturated with bromin, alcohol added, and the mixture distilled. It is a clear, colorless liquid, of a sweet taste, a pleasant smell like that of chloroform, and coloring brown on contact with air and light. Specific gravity 2.834 at  $0^\circ \text{C}$ ., 2.775 at  $15^\circ \text{C}$ ., which is higher than in any other organic compound. Slightly volatile at ordinary temperatures; boils at  $152^\circ \text{C}$ .; melting-point  $-8^\circ \text{C}$ ., and solidifies at  $-9^\circ \text{C}$ . to a crystalline mass. Very slightly soluble in water, but soluble in all proportions in alcohol, ether, benzene, petroleum benzin, glycerin, and in the fixed and volatile oils. It is not inflammable, but when vaporized by the application of heat, its vapor may be burned.

(1) If 10 c.c. (162 minims) of Bromoform be well shaken with 10 c.c. (162 minims) of distilled water and the liquids, upon standing, be allowed to separate completely, the water removed from the layer of Bromoform should be neutral to blue litmus paper (absence of *free acid*), and a portion should not produce a turbidity when treated with silver nitrate T.S. (absence of *bromides* and *brominated compounds*); and another portion treated with potassium iodid T.S. should not be tinted blue upon the addition of starch T.S. (absence of *free bromin*).

### Test for Brucin, $\text{C}_{23}\text{H}_{26}\text{N}_2\text{O}_4 + 4\text{H}_2\text{O}$ .

Brucin, an alkaloid obtained from *nux vomica*, crystallizes in colorless needles; odorless; intensely bitter; readily soluble in alcohol and chloroform, less so in ether, slightly in ammonia; soluble in 150 parts of boiling water, insoluble in fixed caustic alkalies; has an alkaline reaction to red litmus paper.

(1) Reagent 17 does not give a precipitate in a watery solution of Brucin; Reagent 6 only a slight turbidity; Reagents 12, 16, and 24 give slight precipitates.

(2) Brucin imparts to chlorin-water (Reagent 3), when agitated with it, a red color; this color is not permanent.

(3) A purple color, soon fading, forms if one drop of Reagent 22 is spread out as a thin film and one drop of Brucin solution dropped on it; reappears with more Brucin solution.

(4) Brucin dissolves in nitric acid (Reagent 15) with a blood-red color, permanent for days if the alkaloid is present in large excess; the red color fades to yellow when the solution is warmed for ten minutes.

(5) Evaporate solution (4) to dryness; add a trace of sulfuric acid (Reagent 21) and a drop of nitric acid (Reagent 15) to the residue; then add freshly prepared stannous-chlorid solution; the yellow color changes to violet.

(6) If 0.001 gm. ( $\frac{1}{64}$  gr.) potassium dichromate is added to 1 c.c. (16 minims) glacial acetic acid, 0.001 gm. ( $\frac{1}{64}$  gr.) Brucin further added, and sulfuric acid (Reagent 21) dropped into the mixture, a color reaction similar to the nitric-acid reaction is obtained.

(7) Brucin is decomposed by mercurous nitrate (Millon's reagent). Spread one drop of the reagent out to a thin film on a warm porcelain lid, add 0.002 gm. ( $\frac{1}{32}$  gr.) Brucin and 5 drops of water; black mercurous oxid ( $\text{Hg}_2\text{O}$ ) is thereby formed; the Brucin is also colored black. Along the edges the mixture becomes of a dirty-violet or reddish-yellow color if slowly rotated.

*Millon's reagent* is prepared by adding mercury in excess to nitric acid of 1.2 sp. gr. and warming after the action has ceased.

(8) If 0.005 gm. ( $\frac{1}{12}$  gr.) of sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) is added to about 10 c.c. (162 minims) of the yellow solution of Brucin in diluted nitric acid,

there is obtained a brown-colored mixture, changing to a beautiful red and violet.

**Test for Caffein,  $C_5H(CH_3)_3N_4O_2 + H_2O$ .**

*Trimethylxanthin.*

Caffein, the active principle of *coffee*, is in fleecy masses of long white flexible needles with a silky lustre; is odorless; has a bitter taste; very soluble in boiling alcohol and boiling water, less so in chloroform, cold alcohol, and cold water; sparingly in ether and in carbon disulfid; neutral to litmus paper.

(1) A few cubic centimeters of tannin solution (Reagent 24) gives an abundant precipitate in 1 c.c. (16 minims) of Caffein solution; soluble in excess of the reagent. A mixture of one drop of Caffein solution and 40 drops of tannin solution (Reagent 24) is clear.

(2) A few drops of a Caffein solution added to Reagent 22 becomes green after many hours.

(3) To an almost invisible residue of an evaporation of a pure Caffein solution (identity reaction for a fl. extr. of kola-nuts, guarana, coffee, or tea) add a few drops hydrochloric acid (Reagent 5) and a minute crystal of potassium chlorate; evaporate to dryness; cool; expose to vapors of ammonia, when a purple color will be produced.

(4) To a similar residue as under (3) add a little bromin-water (Reagent 2); evaporate to dryness; then add ammonia-water (Reagent 1); a purple color will be obtained.

(5) To 1 c.c. (16 minims) acetic acid (1.064 sp. gr.) add 0.001 gm. ( $\frac{1}{64}$  gr.) Caffein and 0.001 gm. ( $\frac{1}{64}$  gr.) potassium bromate ( $KBrO_3$ ); heat to the boiling-point; it remains colorless. Evaporated to dryness, the mixture leaves a dark-red crystalline residue; heated carefully over the flame, the color becomes more intensely red.

### Test for Cantharidin, $C_{10}H_{12}O_4$ .

Cantharidin is a white substance, in the form of crystalline scales, of a shining, micaceous appearance; inodorous; tasteless; almost insoluble in water and cold alcohol; easily soluble in chloroform, acetone, formic acid, sulfuric acid, acetic acid, the oils, hot alcohol, and ether, which also dissolves it more freely hot than cold; has an acid reaction to very sensitive litmus paper; is volatile at  $100^{\circ}C$ .

(1) The most important reaction of Cantharidin is its vesicating power. Dissolve 0.001 gm. ( $\frac{1}{64}$  gr.) Cantharidin in alcohol, or in an oil, and apply the solution with a camel's-hair brush to the soft part of the upper arm or behind the ear; it blisters.

(2) Cantharidin slowly changes Reagent 22 to a green-colored fluid.

(3) Barium-water gives a crystalline precipitate in a watery boiling solution of Cantharidin, soluble in acetic acid.

(4) Subacetate of lead gives a slight turbidity in a boiling watery solution of Cantharidin.

### Test for Carbolic Acid—Phenol, $C_6H_5(OH)$ .

Carbolic Acid, a constituent of coal-tar, is a white or colorless crystalline mass composed of long needles; when exposed to the light and air it turns pink or brown and liquefies; has a sweetish, aromatic, smoky odor, recalling that of creosote; a sweet burning taste and caustic action on the skin; coagulates albumen and dissolves gelatin; is soluble in water 1:14, with a slightly acid reaction; easily soluble in alcohol, ether, chloroform, benzene, glycerin, carbon disulfid, acetic acid, caustic alkalies, fixed and volatile oils; is inflammable, burning with a reddish flame; melted Carbolic Acid makes filter-paper trans-

parent; it evaporates, however, entirely when slightly warmed.

(1) A green fluid is obtained when 1 c.c. (16 minims) ferric-chlorid solution (Reagent 4) is added to a solution of 20 parts of Carbolie Acid in 10 parts of alcohol of 94 per cent; this solution remains clear after the addition of water, and colors permanent violet if the dilution does not exceed 2000 parts of water to 1 part of Carbolie Acid. Ferric-chlorid solution (Reagent 4) also gives the same violet color reaction in a very dilute (1:3000) watery solution.

(2) A freshly prepared solution of potassium ferricyanid (Reagent 18) gives a blue precipitate in the violet watery fluid obtained above; the Carbolie Acid has reduced the ferric salt to a ferrous compound.

(3) Lead-acetate solution, 10 per cent, gives a voluminous amorphous precipitate in a saturated watery solution of Carbolie Acid.

(4) Carbolie Acid dissolves colorless in sulfuric acid (Reagent 21) in the cold; a red color is developed when the solution is warmed.

(5) Carbolie Acid dissolves in nitric acid (Reagent 15) with a brown-red color.

(6) A permanent blue or bluish-green color reaction takes place when bromin-vapors fall upon a mixture of 5 c.c. (81 minims) of ammonia-water (Reagent 1) and 50 c.c. (1 $\frac{3}{4}$  f. oz.) of a watery solution of Carbolie Acid; this blue color is insoluble in ether and in chloroform; chloroform becomes red-tinged when agitated with the mixture; the blue solution is soluble in alcohol with a green color; a red residue is obtained, changing to blue, by addition of ammonia-water (Reagent 1) when this alcoholic solution is evaporated to dryness; acids change the blue color to red, in which condition it is soluble in ether; ammonia-water (Reagent 1) causes the blue color to appear again.

(7) A pine splinter soaked in a watery solution of Carbolie Acid and dipped into diluted hydrochloric acid becomes of a deep-blue color when exposed for a few moments to the sun's rays; the color firmly resists the action of chlorin; under its influence the blue takes a lighter shade, but soon regains its depth, when the splinter is redipped in the diluted hydrochloric acid.

(8) Carbolie Acid with ammonia-water (Reagent 1) and chlorinated lime ( $\text{CaOCl}_2$ ), or solution of chlorinated soda ( $\text{NaOCl}_2$ ), produce a blue liquid, turned red by acids.

(9) Solutions of Carbolie Acid turn red when boiled with Millon's reagent (see Brucin (7)); if the change does not occur, it may require the addition of a few drops of nitric acid (Reagent 15).

(10) To detect Carbolie Acid in salicylic acid boil 10 grs. of the substance to be tested in half an ounce of water, cool, decant the solution, and add to it 1 minim of a saturated solution of potassium bicarbonate ( $\text{KHCO}_3$ ), 1 minim of anilin, and 5 drops of solution of chlorinated lime ( $\text{CaOCl}_2$ ), when, if Carbolie Acid be present, a deep-blue color is produced.

(11) Bromin-water (Reagent 2) added in excess to a weak solution of Carbolie Acid produces a flocculent, white precipitate. This precipitate is so insoluble that it separates even in the most dilute solutions and affords an extremely delicate test.

#### Test for Carpain, $\text{C}_{14}\text{H}_{25}\text{NO}_2$ .

Carpain, an alkaloid obtained from the *Papaw*, is in large colorless crystals; very bitter; slightly soluble in water, very soluble in chloroform, alcohol, and benzene, much less in ether and in ligroin; is alkaline to red litmus paper.

(1) Bromin-water (Reagent 2) gives a strong turbidity in a cold watery solution of Carpain.

(2) Iodin in potassium iodid (Reagent 7) gives a heavy precipitate with Carpain.

(3) A small particle of a crystal of Carpain dissolved in Reagent 22 gives at first a dirty, not very well-defined color reaction, which develops slowly into a *delicate*, clear, bluish grass-green color, permanent for more than an hour.

(4) A small particle of Carpain dissolved in Reagent 23 gives a *delicate* lilac color, lasting for more than an hour, and fading entirely to a colorless fluid.

(5) A solution of Carpain in carbon disulfid slowly colors yellow.

### Test for Chloral Hydrate, $\text{CCl}_3\text{CH}(\text{OH})_2$ .

Chloral Hydrate is in white transparent crystals; has a pungent but agreeably aromatic odor; a bitter, astringent, slightly caustic taste; is easily soluble in water, alcohol, ether, volatile and fatty oils, liquid Hydrocarbons, chloroform, carbon disulfid, oil of turpentine, etc.—in fact there seems to be no fluid in which Chloral Hydrate is not soluble. Its aqueous solution is neutral to litmus paper. Exposed to the air it slowly volatilizes, and, like camphor, when enclosed in a bottle, covers the interior surface with numerous minute crystallizations.

(1) Camphor, phenol, thymol, and menthol all liquefy when triturated with Chloral Hydrate in equal parts.

(2) Crystals of Chloral Hydrate sink in sulfuric acid, but soon rise to the surface, however, and liquefy, decomposing into a turbid, colorless top layer of metachloral and chloral.

(3) Litmus gives an acid reaction, and silver-nitrate solution, 5 per cent, a voluminous precipitate, in water

boiled with a small lump of zinc and a trace of Chloral Hydrate. Silver-nitrate solution produces only a slight turbidity in freshly prepared watery or alcoholic solutions of Chloral Hydrate.

(4) A solution of 0.020 gm. ( $\frac{1}{3}$  gr.) Chloral Hydrate, 0.030 gm. ( $\frac{1}{2}$  gr.) resorcin, and five drops caustic-soda solution (Reagent 20) has an intense-red color, changing to brown-red; this solution has a yellowish-green fluorescence when diluted with 100 c.c. ( $3\frac{1}{2}$  f. oz.) water; acids destroy the fluorescence. When 0.010 gm. ( $\frac{1}{8}$  gr.) resorcin is taken instead, the color is merely pink and fades slowly to greenish yellow. By substituting lime-water for the caustic-soda solution, the fluid is lilac at first, but finally turns to a yellowish green.

(5) If equal parts Chloral Hydrate, resorcin, and slacked lime are triturated together, a white powder is obtained that soon becomes red (intensely red when moistened) and finally turns brown. When thymol is substituted for resorcin a beautiful violet powder is obtained.

(6) If 0.010 gm. ( $\frac{1}{8}$  gr.) Chloral Hydrate be dissolved in 1 c.c. (16 minims) sodium-hydrate solution (Reagent 20) a turbid fluid is obtained, soon giving off the odor of chloroform, the latter falling to the bottom in small drops.

(7) Mixed with a 5 per cent solution of carbolic acid, and an equal bulk of sulfuric acid (Reagent 21) added, Chloral Hydrate gives a pink color.

### Test for Chrysarobin, $C_{30}H_{26}O_7$ .

Chrysarobin, a principle obtained from *Goa powder*, is a pale orange-yellow powder; tasteless and odorless; slightly soluble in cold water, ether, or alcohol; more soluble in boiling alcohol, in solutions of the alkalies, in chloroform, benzene, and glacial acetic acid; the watery solution is neutral to litmus paper.

(1) Caustic-soda solution (Reagent 20) changes the yellow color of the opalescent filtrate of Chrysarobin, when this has been boiled with water, to red; this watery solution is neutral.

(2) Ferric-chlorid solution (Reagent 4) produces in the alcoholic solution of Chrysarobin a dirty green-brown color.

(3) Caustic potash and soda dissolve Chrysarobin with a red color, which gives a green fluorescent fluid when diluted with water or alcohol.

(4) 0.010 gm. ( $\frac{1}{6}$  gr.) Chrysarobin shaken with 50 c.c. ( $1\frac{1}{2}$  f. oz.) lime-water gives to the latter a violet-red color.

(5) Chrysarobin shaken with ammonia-water (Reagent 1) produces a brown-red color; warming deepens the color.

(6) Sulfuric acid (Reagent 21) immediately dissolves Chrysarobin with a yellow-red color; nitric acid (Reagent 15) slowly, with a faint yellow color.

(7) Triturate 0.005 gm. ( $\frac{1}{40}$  gr.) Chrysarobin and 0.005 gm. ( $\frac{1}{40}$  gr.) sodium nitrate ( $\text{NaNO}_3$ ) with a glass rod barely moistened by means of a damp cloth or paper, so that the mixture of Chrysarobin and sodium nitrate will remain nearly dry. Spread the mixture as thinly as possible on a porcelain lid with a glass rod to which only a trace of sulfuric acid (Reagent 21) clings. Strew on this layer a few small particles of solid caustic potash or soda; a black or dark-violet zone forms around each, as moisture is attracted from the atmosphere, until soon the particles of potash or soda are so many black drops. The layer becomes violet when ammonia-water (Reagent 1) is substituted for the fixed alkalies. This is the most characteristic reaction of Chrysarobin.

**Test for Cinchonidin Sulfate,  $(C_{19}H_{22}N_2O)_2H_2SO_4 + 6H_2O$ .**

Cinchonidin Sulfate is in white, silky, acicular crystals; odorless; having a very bitter taste; soluble in water and alcohol; less so in chloroform; almost insoluble in ether; neutral or faintly alkaline to litmus paper. Heated in a glass tube, Cinchonidin Sulfate yields a beautiful red-colored tar. This test can be made use of to distinguish Peruvian from any other bark; only cinchona alkaloids give this reaction.

(1) 1 c.c. (16 minims) of a 5 per cent potassium-iodid solution gives a dense turbidity in 1 c.c. (16 minims) of a watery solution of Cinchonidin Sulfate.

(2) Dissolve 0.50 gm. (8 grs.) of Cinchonidin Sulfate in 15 c.c. (243 minims) of alcohol (83 sp. gr.) diluted with 5 c.c. (81 minims) of water and acidulated with 2 c.c. (32 minims) of 10 per cent sulfuric acid; add to this solution a solution of 0.20 gm. (3 grs.) iodine in 10 c.c. (162 minims) of alcohol (83 sp. gr.); warm the mixture slightly and allow to cool; a *yellow* crystalline precipitate is formed, turning brown when dry.

(3) A precipitate is obtained when lime-water (Reagent 10) is added to 1 c.c. (16 minims) of the cold saturated solution of Cinchonidin Sulfate, but it dissolves again when a total amount of 13 c.c. (211 minims) of lime-water is added.

(4) Dissolve 0.50 gm. (8 grs.) of the Cinchonidin Sulfate in 20 c.c. (324 minims) water by slightly warming; add 1.50 gm. (23 grs.) finely pulverized Rochelle salt (potass. et sod. tartras); dissolve and cool; a crystalline precipitate of cinchonidin tartrate, sparingly soluble, is obtained.

(5) Dissolve 0.010 gm. ( $\frac{1}{10}$  gr.) Cinchonidin Sulfate in 10 c.c. (162 minims) water. The precipitate caused by adding ammonia-water (Reagent 1) to 5 c.c. (81 minims)

of this solution does not dissolve in 30 c.c. (1 f. oz.) ammonia-water (Reagent 1). To the other 5 c.c. (81 minims) add ammonia-water (Reagent 1) to obtain a precipitate; then add 5 c.c. (81 minims) ether; agitate strongly; the precipitate immediately dissolves.

(6) Add concentrated sulfuric acid (Reagent 21) to a small quantity of Cinchonidin Sulfate, and then a crystal of dichromate of potassium; a yellowish-green color is produced, which gradually changes to grass-green.

**Test for Cinchonin Sulfate,  $(C_{19}H_{22}N_2O)2H_2SO_4 + 2H_2O$ .**

Cinchonin Sulfate is in short, white, lustrous, prismatic, hard crystals; without odor, having a very bitter taste; soluble in water, alcohol, and chloroform; insoluble in ether; neutral to litmus paper; yields a red tar when heated in a glass tube.

(1) A saturated watery phenol solution causes a strong turbidity in a saturated watery solution of Cinchonin Sulfate.

(2) The reaction with potassium iodid does not differ from the one described for cinchonidin sulfate.

(3) If chlorin-water (Reagent 3) is poured on some Cinchonin Sulfate, this colors faintly yellow only in the course of a day; the reaction takes place much quicker with cinchonidin, quinidin, quinin, or their salts.

(4) Dissolve 0.010 gm. ( $\frac{1}{8}$  gr.) Cinchonin Sulfate in 10 c.c. (162 minims) water. The precipitate caused by ammonia-water (Reagent 1) added to 5 c.c. (81 minims) of this solution is not dissolved, even by adding as much as 50 c.c. ( $1\frac{1}{2}$  f. oz.) of the reagent. To the other 5 c.c. (81 minims) of the solution add ammonia-water (Reagent 1) to obtain a precipitate; then add 10 c.c. (162 minims) ether, and agitate strongly. The precipitate does not go into solution.

**Test for Cocain Hydrochlorate,  $C_{17}H_{21}NO_4HCl$ .**

Cocain Hydrochlorate is the hydrochlorate of an alkalioid obtained from *Coca*. It occurs in colorless, transparent, flaky, lustrous crystals or a white crystalline powder of a saline, slightly bitter taste, and producing upon the tongue a tingling sensation followed by numbness; applied to the eye it causes dilatation of the pupil. It is permanent in the air; soluble in half its weight of water, in alcohol, and in chloroform; almost insoluble in benzene, petroleum benzin, and ether; its solution is neutral to litmus paper. Melting-point  $200-202^{\circ}C.$ , when the liquid gives off a fruit-like odor; it turns brown if heated higher, and leaves no residue on incineration.

(1) Lime-water (Reagent 10) gives a turbidity in the watery solution of Cocain Hydrochlorate (1:50) which is redissolved when about 10 c.c. (162 minims) of the reagent is added; less ammonia-water (Reagent 1) is required to redissolve it.

(2) 0.10 gm. ( $1\frac{1}{2}$  grs.) Cocain Hydrochlorate dissolves without color in 1 c.c. (16 minims) of sulfuric acid (Reagent 21), but the solution turns brown when heated.

(3) When Cocain Hydrochlorate is triturated with an equal quantity of calomel, the mixture blackens by the slightest humidity (breathing upon it) or by moistening with alcohol.

(4) 0.10 gm. ( $1\frac{1}{2}$  grs.) Cocain Hydrochlorate changes the color of 1 c.c. (16 minims) of sulfuric acid containing chromic acid (Reagent 22) to red when strewn upon it; the change to green (owing to formation of chromium sulfate) does not commence under an hour.

(5) A faint-yellow fluid is obtained, which becomes red by boiling, when one drop of ferric-chlorid solution (Reagent 4) is added to a solution of 0.020 gm. ( $\frac{1}{2}$  gr.) of Cocain Hydrochlorate in 2 c.c. (32 minims) of water.

This red compound (benzoate of iron) is insoluble in ether and chloroform, but soluble in alcohol; the red-colored fluid becomes yellow on the addition of hydrochloric acid (Reagent 5).

(6) A green-colored fluid is obtained when 1 c.c. (16 minims) sulfuric acid (Reagent 21) is heated with 0.010 gm. ( $\frac{1}{8}$  gr.) Cocain Hydrochlorate until vapors are seen, and 0.020 gm. ( $\frac{1}{4}$  gr.) potassium iodate is now dropped into the warm solution; the fluid changes to a red-brown if overheated.

(7) Evaporate to dryness on a water-bath a solution of 0.010 gm. ( $\frac{1}{8}$  gr.) Cocain Hydrochlorate in 1 c.c. (16 minims) of nitric acid (Reagent 15); the residue is colorless. Add to it a few particles of caustic potash or soda and a few drops of alcohol, when a delicious and very permanent odor of benzoic ethyl ester is developed.

### Test for Codein, $C_{17}H_{17} \cdot OCH_3(OH)NO + H_2O$ .

Codein, an alkaloid of opium, is in white or nearly translucent octahedral crystals; odorless; has a bitter taste; easily soluble in chloroform, ether, alcohol, ammonia-water, and carbon disulfid; less soluble in water; in boiling-water Codein forms oily drops; the watery solution has an alkaline reaction to litmus paper.

(1) Iodin solution (Reagent 6) causes a turbidity in a watery solution of Codein (1:80), soon clearing up but re-appearing on addition of more of the reagent.

(2) 0.050 gm. ( $\frac{3}{4}$  gr.) Codein colors red when strewn upon 2 c.c. (32 minims) nitric acid (Reagent 15); the acid itself becomes yellow colored only when warmed.

(3) Dissolve 0.075 gm. ( $1\frac{1}{8}$  grs.) potassium ferricyanid ( $K_6Fe_2(CN)_{12}$ ) in 200 c.c. ( $6\frac{3}{4}$  f. oz.) of water, to which is added 1 c.c. (16 minims) of Reagent 4; put a few drops of

this mixture on a porcelain lid, and strew on it a small quantity of Codein; it assumes a brown color.

(4) Concentrated sulfuric acid (Reagent 21) dissolves Codein colorless, even when warmed five minutes with the acid on the water-bath; the acid solution becomes immediately green, blue, and violet when touched, yet warm, with a very dilute solution of ferric chlorid on a small glass rod and kept warm on the water-bath for a little while.

(5) The solution of Codein in sulfuric acid (Reagent 21) becomes green, changing to dark violet when stirred with a glass rod the size of a match, slightly moistened with a trace of nitric acid (Reagent 15).

(6) A mixture of Codein and sodium nitrate strewn on sulfuric acid (Reagent 21) gives a brownish-black coloration, changing to green and red-brown.

(7) Codein remains colorless when agitated with weak chlorin-water (Reagent 3) diluted one-half; strong chlorin-water gives a beautiful red color, especially when ammonia-water (Reagent 1) is added.

(8) Diluted sulfuric acid (1.11 sp. gr.) warmed with Codein on a water-bath becomes pink-colored.

(9) Codein leaves a dark-brick-red colored mass when a crystal is dissolved in nitric acid (Reagent 15), the solution evaporated to dryness and a little of a solution of potassium hydrate in absolute alcohol (1:10) poured over it, and the mixture evaporated again.

(10) 0.020 gm. ( $\frac{1}{3}$  gr.) Codein triturated with 0.010 gm. ( $\frac{1}{6}$  gr.) cane-sugar and two drops sulfuric acid (Reagent 21) gives a rose-red mixture.

#### Test for Colchicin, $C_{22}H_{25}NO_6$ .

Colchicin is a yellowish, amorphous powder; without odor; very bitter, soluble in water, diluted acids, and

alkaline solutions; very soluble in alcohol and chloroform; hardly soluble in ether, and insoluble in benzene. The watery and alcoholic solutions have no reaction on litmus paper.

(1) A piece of potassium nitrate, added to a solution of Colchicin in sulfuric acid, produces a beautiful blue color, changing to green, dark brown, or purple, and finally reddish yellow.

(2) Evaporate 0.5 c.c. (8 minims) of a solution of Colchicin by rotating over a large surface in a warm evaporating-dish; a few drops of a ten times diluted ferric-chlorid solution (Reagent 4) added to the brown-yellow film, previously moistened with 0.5 c.c. (8 minims) alcohol, produces a greenish-brown color; sulfuric acid (Reagent 21) colors the film bright yellow; violet streaks, soon fading to brown, are obtained by touching the sulfuric acid with a small glass rod moistened with a trace of nitric acid (sp. gr. 1.4); nitric acid (Reagent 15) alone gives the same violet color reaction.

(3) A strong watery solution of pure phenol gives, with a watery Colchicin solution, a white precipitate, turning bright yellow.

(4) 5 c.c. (81 minims) chlorin-water (Reagent 3) gives a yellow precipitate in 0.5 c.c. (8 minims) of a Colchicin solution; soluble in ammonia-water (Reagent 1) with an orange color.

### **Test for Coniin, $C_8H_{16}NH_6$ .**

Coniin is a yellowish, oily, volatile, liquid alkaloid; floats on water; has a strong, repulsive, penetrating odor compared to that of the urine of mice; an acrid taste suggestive of tobacco; is freely soluble in alcohol, ether, and the fixed and volatile oils; slightly soluble in water (1:150); the watery solution has an alkaline reaction on litmus paper.

(1) 0.050 gm. ( $\frac{3}{4}$  gr.) of calomel blackens when shaken with 5 c.c. (81 minims) of Coniin solution.

(2) Add to ten drops of ether in a shallow glass crystallizing-dish two drops of Coniin and cover with filter-paper; set upon the paper a common-size watch-crystal containing bromin-water (Reagent 2); invert a beaker over the whole arrangement; needle-shaped crystals of Coniin hydrobromate soon form in the dish, as well as next to the watch-crystal. If a watery solution of this Coniin salt is shaken with magnesia and next with carbon disulfid, the latter turns yellow.

(3) A blue precipitate is formed when one drop of a 10 per cent copper-sulfate solution is dropped into five drops of the watery solution of Coniin.

(4) Carbon disulfid colors yellow when shaken with a watery Coniin solution (1:150).

(5) Coniin dissolves sulfur. If 0.020 gm. ( $\frac{1}{3}$  gr.) of sulfur is moistened with Coniin, it softens and colors red.

(6) Warmed with potassium dichromate and sulfuric acid (Reagent 21), Coniin yields butyric acid, detected by its odor of rancid butter.

### Test for Corrosive Sublimate, $\text{HgCl}_2$ .

#### *Corrosive Mercuric Chlorid.*

Corrosive Sublimate is in heavy, white, opaque, or translucent crystalline masses; is odorless, and has a sharp, metallic taste; volatilizes in dense, white vapors, leaving no residue; soluble in water, alcohol, ether, and glycerin; the aqueous solution reddens blue litmus paper.

(1) Acidulate the liquid to be tested with hydrochloric acid (Reagent 5), and pass through it an excess of hydrogen sulfid ( $\text{H}_2\text{S}$ ); a black precipitate, insoluble in ammonium sulfid and in nitric acid, indicates Corrosive Sublimate.

(2) The fixed alkalies and alkaline earths produce a yellow precipitate of mercuric oxid ( $\text{HgO}$ ) in solutions of Corrosive Sublimate; this oxid is produced in the process of preparing yellow wash, which is obtained by mixing Corrosive Sublimate with lime-water.

(3) Potassium iodid yields a red precipitate when added to solutions of Corrosive Sublimate; the precipitate is soluble in excess both of the precipitant and the mercuric salt.

(4) Place a small piece of bright copper, about half an inch long and a quarter of an inch broad, in a solution of any salt of mercury and heat in a test-tube; the copper becomes coated with mercury in a state of fine division; pour away the supernatant liquid from the copper; wash the latter once or twice by pouring water into and then out of the test-tube; remove the copper, take off excess of water by gentle pressure in a piece of filter-paper, dry the copper by passing it quickly through a flame, holding it by the fingers; finally place the copper in a dry, narrow test-tube and heat to redness in a flame, the test-tube being held almost horizontally; the mercury sublimes and condenses as a whitish film of minute globules on the cool part of the test-tube beyond the flame; the globules aggregate on gently pressing with a glass rod, and are especially visible where flattened between the rod and the side of the test-tube. This is a very delicate test and eliminates mercury in presence of other substances, organic and inorganic.

#### Test for Cotoin, $\text{C}_{22}\text{H}_{18}\text{O}_6$ .

Cotoin, the active principle of coto bark, is in yellowish-white, light, prismatic crystals, resembling commercial gallic acid; has a sharp taste; without odor; soluble in alcohol, ether, and chloroform; less soluble in carbon

disulfid, almost insoluble in water; neutral to litmus paper.

(1) Ferric-chlorid solution (Reagent 4) gives, in a boiling saturated watery solution of Cotoin, a voluminous brown precipitate; the fluid becomes dark red-brown at first.

(2) A 10 per cent lead-acetate solution gives a yellow turbidity in a watery solution of Cotoin.

(3) Sulfuric acid (Reagent 21) changes the color of a solution of Cotoin in ether or chloroform, by shaking, to a brown-yellow.

(4) Ferric-chlorid solution (Reagent 4) colors the alcoholic solution of Cotoin red-brown.

(5) Cotoin is soluble in ammonia-water (Reagent 1) and lime-water (Reagent 10) with a yellow color.

(6) Cotoin strewn on sulfuric acid (Reagent 21) colors the latter greenish yellow; an addition of sodium nitrite ( $\text{NaNO}_2$ ) gives a dark-red color.

(7) Cotoin does not give a color reaction at first with nitric acid (Reagent 15), but in the course of an hour a permanent pink hue is obtained.

### Test for Creosote.

Creosote is an oily liquid, colorless when fresh, but, as met with in commerce, frequently of a brownish tinge; has a burning taste and a penetrating, disagreeable odor, like that of smoked meat; applied to the skin it destroys the cuticle, and leaves a white spot; it is neutral to litmus paper; is inflammable; very slightly soluble in cold water, in which it sinks; insoluble in glycerin (distinction from liquid carbolic acid); soluble, in all proportions, in alcohol, ether, chloroform, benzin, carbon disulfid, acetic acid, and fixed and volatile oils.

(1) The watery solution of Creosote gives a green color

with ferric-chlorid solution (Reagent 4), rapidly changing to a reddish-brown precipitate.

(2) The filtrate from a hot watery solution of Creosote yields a reddish-brown precipitate with bromin-test solution (distinction from carbolic acid).

(3) An alcoholic solution of iron perchlorid added to an alcoholic solution of Creosote produces a deep greenish-blue color, but with carbolic acid a light brown.

### **Test for Cryptopin, $C_{21}H_{23}NO_5$ .**

Cryptopin, an alkaloid from opium, is in small, colorless, odorless, tufted crystals, or in a crystalline powder; soluble in acetone, benzene, and chloroform; sparingly soluble in alcohol; hardly at all in ether, carbon disulfid, or water; has a bitter taste, followed by a cooling sensation in the mouth, like that produced by peppermint, or a choking feeling in the throat; has a strong alkaline reaction.

(1) Chlorin-water (Reagent 3) dissolves Cryptopin with a yellow color, increasing in intensity to red on the addition of ammonia-water (Reagent 1).

(2) Sulfuric acid (Reagent 21) colors a hardly visible particle of Cryptopin yellow at first, soon changing to red; soon pink-colored streaks flow from the fragments when strewn upon the acid, which assume a violet color, changing to blue and green; the color is blackish blue when a trace of ferric-chlorid solution (Reagent 4) has been added to the acid; if a small particle of Cryptopin is triturated with a microscopical crystal of sodium nitrite ( $NaNO_2$ ) and then thrown on the sulfuric acid (Reagent 21), it gives a dark violet color changing to green, and on the addition of an excess of water to red.

(3) Cryptopin thrown upon Reagent 22 immediately colors the latter green.

(4) Cryptopin becomes yellow-colored when treated with

sulfuric acid (Reagent 21) and bismuth subnitrate; the yellow color changes to a pure green, afterwards turning black; with molybdic acid the color changes to green. black and blue. with titanio acid, yellow, brown, and pink; with tungstic acid, yellow-brown and greenish gray.

(5) Cryptopin throws down a blue precipitate in a mixture of potassium-ferricyanid and ferric-chlorid solutions after a few minutes.

(6) If Cryptopin hydrochlorate be dissolved in about 30 parts of hot water and set aside, instead of crystallizing it forms a jelly closely resembling that of pure gelatin.

### Test for Cumarin, $C_9H_6O_2$ .

Cumarin is in large, colorless crystals; has a strong, agreeable, aromatic odor and a sharp, bitter taste; soluble in alcohol, ether, and chloroform; much less soluble in carbon disulfid and in water; does not change litmus paper.

(1) If 0.010 gm. ( $\frac{1}{8}$  gr.) Cumarin is dissolved in 1 c.c. (16 minims) sulfuric acid (Reagent 21) and 0.010 gm. ( $\frac{1}{8}$  gr.) sodium nitrate ( $NaNO_3$ ) is added to the solution, this becomes red, which color commences in the test-tube from above in the course of a few hours; saturated with caustic-soda solution (Reagent 20) the color changes to pure yellow.

(2) A mixture of 0.020 gm. ( $\frac{1}{3}$  gr.) Cumarin and 1 gm. ( $15\frac{1}{2}$  grs.) caustic potash turns yellow when warmed; this color disappears on further heating; it gives the violet salicylic-acid reaction with ferric-chlorid solution (Reagent 4) if dissolved in slight excess of diluted sulfuric acid (sp. gr. 1.11) and one drop of Reagent 4 is added to the cooled solution.

**Test for Cuprein,  $C_{19}H_{22}N_2O_2$ .**

Cuprein is in small crystals; soluble in alcohol; sparingly soluble in ether, benzene, chloroform, or carbon disulfid; these solutions quickly turn brown; alkaline to litmus paper.

(1) The alcoholic solution of Cuprein becomes blood-red on addition of ferric-chlorid solution (Reagent 4).

(2) Add to 1 c.c. (16 minims) of Cuprein solution 1 c.c. (16 minims) chlorin-water (Reagent 3) and then slowly add a few drops ammonia-water (Reagent 1); an emerald-green color is developed. This is the Thalleioquin reaction.

**Test for Digitalin,  $C_5H_8O_2$ .**

Digitalin is a white amorphous powder; odorless; having a bitter taste; soluble in water 1:1000; almost insoluble in ether and chloroform; the watery solution foams when shaken; soluble in cold alcohol 1:100, and readily so in hot alcohol; if a small quantity of alcohol is used in the latter test, a kind of jelly will form on cooling.

(1) Digitalin is thrown down as an amorphous precipitate if ether is added to an alcoholic solution, and the filtrate will yield a similar precipitate when another portion of ether is added.

(2) Digitalin dissolves with a yellow color in hydrochloric acid (Reagent 5) and in sulfuric acid (Reagent 21); the solutions soon turn red.

(3) A bluish-red color reaction is obtained if 1 drop of nitric acid (Reagent 15), 1 drop of ferric-chlorid solution (Reagent 4), or 1 drop of bromin-water (Reagent 2) be added to the yellow-colored solution of Digitalin in sulfuric acid (Reagent 21).

(4) An intense red color is produced if a trace of Digitalin dissolved in water is mixed with a weak aqueous

solution of inspissated bile and sufficient sulfuric acid (Reagent 21) added to raise the temperature to 70° C.

### Test for Diuretin.

Diuretin, the salicylate of sodium and theobromin, is a white crystalline powder; at first sweetish, later salty, finally of a bitter taste; soluble in warm water 1:4; has an alkaline reaction to litmus paper.

(1) To 2 c.c. (32 minims) of a saturated aqueous solution of Diuretin add 1 drop of tannin solution (Reagent 24); an abundant precipitate is thrown down, soluble in a Diuretin solution as well as in an excess of the reagent.

### Test for Ecgonin, $C_9H_{15}NO_3 + H_2O$ .

Ecgonin, an alkaloid obtained from coca, is in monoclinic crystals; when heated they give off vapors of an alkaline reaction and not disagreeable odor; easily soluble in water; less soluble in chloroform; much less in alcohol, ether, or carbon disulfid; a watery solution does not affect litmus paper; has a slight bitter taste, without the peculiar choking and numbness produced by cocaine.

(1) Ecgonin darkens the color of a diluted (1:2) ferric-chlorid solution (Reagent 4) when dissolved therein.

(2) Ecgonin soon causes a green color reaction in Reagent 22.

### Test for Emetin, $C_{30}H_{40}N_2O_5$ .

Emetin, the active principle of ipecac, is a yellowish-white amorphous powder; odorless; nearly insoluble in cold water, slightly so in warm; easily soluble in alcohol, ether, chloroform, and carbon disulfid; on red litmus paper it gives an alkaline reaction if moistened with water or alcohol; its watery solution has a bitter taste.

(1) 1 c.c. (16 minims) of tannin solution (Reagent 24) gives a slight turbidity in 2 c.c. (32 minims) of a solution of Emetin in boiling water.

(2) A faint, reddish-yellow fluid is obtained in the course of an hour by mixing 1 c.c. (16 minims) of the aqueous solution of Emetin with 1 c.c. (16 minims) hydrochloric acid (Reagent 5) and adding 0.005 gm. ( $\frac{1}{12}$  gr.) potassium chlorate in fine crystalline powder. A variation of this test, immediately active, is had by adding to the Emetin a solution of chlorinated lime in the presence of a trace of acetic acid; this gives a bright-orange to canary-yellow color reaction.

(3) Triturate 0.020 gm. ( $\frac{1}{50}$  gr.) ammonium molybdate with 5 drops sulfuric acid (Reagent 21); Emetin gives a brown color, soon changing to dark green when thrown upon the foregoing and stirred with a glass rod.

(4) Add a few drops of nitric acid (Reagent 15) to a small particle of Emetin; it forms a nitrate, at first bulky and very slightly soluble in water, but soon agglutinating into a brown, pitch-like matter, very soluble in water and uncrystallizable.

### **Test for Eseridin, $C_{15}H_{23}N_3O_3$ .**

Eseridin, an alkaloid obtained from the calabar bean, is in small crystals; sparingly soluble in water, even at the boiling-point; colors the water brownish red at this temperature; easily soluble in chloroform; less so in ether, alcohol, and carbon disulfid; when heated above  $132^{\circ}$  C. it evolves biting, stinging vapors of alkaline reaction; the crystals are neutral.

(1) Eseridin colors bromin-water (Reagent 2) or chlorin-water (Reagent 3) violet-red; ammonia-water (Reagent 1) increases this color.

(2) Eseridin colors nitric acid (Reagent 15) yellow first,

red afterwards; this solution leaves a yellowish-red residue when evaporated to dryness, that changes slowly to green on attracting moisture from the atmosphere.

(3) Ammonia-water (Reagent 1), lime-water (Reagent 10), or caustic-soda solution (Reagent 20), poured on Eseridin, becomes slowly yellow and dirty greenish.

(4) A red solution is obtained by slightly warming and agitating a trace of Eseridin and the smallest particle of potassium iodate ( $\text{KIO}_3$ ), with a few c.c. of water; chloroform extracts a brown-red color from this mixture; if acetic acid is present, however, the chloroform becomes violet.

### Test for Eserin, $\text{C}_{15}\text{H}_{21}\text{N}_3\text{O}_2$ .

#### *Physostigmin.*

Eserin, an alkaloid from the calabar bean, is in colorless or pinkish crystals; slightly soluble in water; readily soluble in alcohol, ether, chloroform, benzene, and carbon disulfid; the watery solution is tasteless, has an alkaline reaction, and darkens to a red and dark-brown fluid even in closed bottles in the dark, when occasionally exposed to light; this coloring of the watery solution takes place immediately when the solution is warmed.

(1) An amorphous residue of a permanent blue color is obtained if a trace of Eserin, or of one of its salts, is evaporated in the presence of an excess of ammonia; this blue alkaloid dissolves in dilute acids with a red color; the solution has a beautiful red fluorescence in reflected light; when evaporated it leaves a residue that is green at first, changing to blue afterwards, the blue residue is soluble in water, alcohol, and chloroform, but not in ether.

(2) When ammonia-water (Reagent 1), lime-water (Reagent 10), or caustic-soda solution (Reagent 20) is poured on Eserin, or one of its salts, it is colored red.

(3) A red fluid is obtained when 0.010 gm. ( $\frac{1}{10}$  gr.) of Eserin, 0.050 gm. ( $\frac{3}{4}$  gr.) of slaked lime, and 1 c.c. (16 minims) of water are added together; warmed in a water-bath it turns green, and a piece of red litmus paper suspended in the test-tube colors blue; the green solution does not lose its color by evaporation.

(4) Eserin thrown upon sulfuric acid (Reagent 21), or on Reagent 22, gives a yellow color reaction.

(5) Nitric acid (Reagent 15) turns Eserin yellow first, red afterwards; the residue of evaporation is yellow-red.

(6) Dissolve 0.010 gm. ( $\frac{1}{10}$  gr.) of an Eserin salt in 10 c.c. (162 minims) of water and drop one or two drops of the solution into the eye; within fifteen minutes the pupil becomes contracted to the size of a pin-head; this physiological experiment causes little inconvenience, and passes off unnoticed.

### Test for Europhen, $2\left(\begin{smallmatrix} \text{C}_4\text{H}_9 \\ \text{C} \text{ H}_3 \end{smallmatrix} \text{C}_6\text{H}_3\text{O}\right)\text{HI}$ .

#### *Di-isobutylorthocresol iodid.*

Europhen is a light, dull-yellow amorphous powder, tasteless, with a faint aromatic odor suggestive of carbolic acid; insoluble in water, caustic potash or soda solutions, glycerin, and in diluted acids; easily soluble in alcohol, ether, chloroform, and fatty oils, with a yellow or reddish-yellow color; water gives a bright-yellow flocculent sediment in an alcoholic solution, neutral to litmus paper.

(1) Concentrated sulfuric acid (Reagent 21) changes Europhen to a dark-brown powder.

(2) 0.50 gm. (8 grs.) Europhen, 5 gms. (77 grs.) zinc-dust, and 20 c.c. (325 minims) water, warmed for an hour on the water-bath with frequent agitation, yields a colorless filtrate, from which carbon disulfid does not extract iodine; these conditions change, however, when the zinc iodid is decomposed by the addition of bromin-water (Reagent 2).

**Test for Exalgin,  $C_6H_5 \cdot NCH_3(C_2H_5O)$** *Methylacetanilid.*

Exalgin forms large crystalline needles; very soluble in alcohol, chloroform, and carbon disulfid; less soluble in ether; soluble in cold water 1:60, in boiling water 1:2; the watery solutions are tasteless and neutral.

(1) Sodium nitrate or nitrite added to a solution of Exalgin in sulfuric acid (Reagent 21) gives yellow to greenish color reactions.

(2) Exalgin at first has no effect upon Reagent 22, but later, however, changes slowly to green.

**Test for Gallic Acid,  $C_6H_2(OH)_3COOH + H_2O$ .**

Gallic Acid crystallizes in slender, silky, fawn-colored needles; odorless, and of a sour, astringent taste, and acid reaction; soluble in water, alcohol, ether, and glycerin; slightly soluble in chloroform and in carbon disulfid; 1 drop of a saturated aqueous solution is nearly tasteless, has an acid reaction, and decomposes sodium hyposulfite solution 1:50 (distinction from tannin).

(1) Pyrogallol sublimes in crystals when 0.050 gm. ( $\frac{3}{4}$  gr.) Gallic Acid, mixed with 0.20 gm. (3 grs.) calcium carbonate, is heated in a glass tube; the sides of the tube cover with moisture at the same time; the mixture changes to a dark-brown powder.

(2) Three drops of a saturated aqueous solution of Gallic Acid soon crystallize when carefully brought as a top layer upon 1 c.c. (16 minims) sulfuric acid (Reagent 21); these crystals dissolve colorless by shaking.

(3) One drop of a saturated aqueous solution of Gallic Acid gives a clear mixture with 1 drop of copper-sulfate solution (1:14) and 1 c.c. (16 minims) of water; a brown

precipitate forms, however, if 0.010 gm. ( $\frac{1}{10}$  gr.) sodium acetate or calcium carbonate is shaken with the mixture.

(4) If 0.010 gm. ( $\frac{1}{10}$  gr.) Gallic Acid be mixed with 0.010 gm. ( $\frac{1}{10}$  gr.) ferrous sulfate and a few cubic centimetres of distilled water (the distilled water must be boiled first and allowed to cool) in a narrow test-tube, even shaking the contents does not cause any immediate coloration; later, however, the solution becomes slowly blue colored, commencing at the top, and, in the course of the day, a black flocculent precipitate settles down therein; the coloring commences very soon if 0.001 gm. ( $\frac{1}{1000}$  gr.) sodium acetate is added in making the reaction.

(5) Gallic Acid does not precipitate morphin or quinin salts from a saturated aqueous solution of the latter, nor gelatin, albumen, or starch (distinction from tannin).

(6) Gallic Acid gives color reactions with alkaline fluids; 2 c.c. (32 minims) of a watery solution will give a yellow-red mixture with 1 c.c. (16 minims) ammonia-water (Reagent 1); poured on calcium carbonate it turns green; gives a colorless mixture with an equal volume of lime-water (Reagent 10), a faint green color developing a few minutes later; this takes place sooner when 3 c.c. (48 minims) of lime-water are taken, but the fluid in this case is darker and changes soon to a bluish hue; a black flocculent sediment forms also.

(7) A deep rose color is developed when a solution of Gallic Acid is treated with potassium cyanid (distinction from tannin).

(8) Gallic Acid produces a deep bluish-black color with ferric-chlorid solution (Reagent 4), which disappears when the solution is heated.

(9) On adding to a cold, saturated, aqueous solution of Gallic Acid an excess of lime-water (Reagent 10) a bluish-white precipitate will form, and the liquid acquires a tint which is blue by reflected light and green by transmitted

light, and becomes pink on the addition of a large excess of the lime-water (distinction from tannin).

(10) If 5 c.c. (81 minims) of a cold saturated aqueous solution of Gallic Acid be treated in a watch-glass with 6 drops of sodium-hydrate solution (Reagent 20), the liquid will gradually acquire a deep-green color, which is changed to reddish or brownish red by acids.

### Test for Gelsemin, $C_{24}H_{28}N_2O_4$ .

Gelsemin is a white amorphous powder; easily soluble in alcohol, ether, and chloroform, scarcely soluble in water, which solution has a very bitter taste; it dilates the pupil.

(1) Add to a small particle of Gelsemin 2 or 3 drops of sulfuric acid (Reagent 21); no reaction follows; it dissolves colorless; add a small quantity of manganese dioxid and stir in with a glass rod; cherry-red streaks flow from the manganese dioxid; the same cherry-red color is obtained by substituting Reagent 23 for the manganese dioxid.

(2) Dissolve 0.002 gm. ( $\frac{1}{32}$  gr.) of Gelsemin in 1 drop sulfuric acid (Reagent 21), add 0.005 gm. ( $\frac{1}{12}$  gr.) finely pulverized sugar to this acid solution; the sugar colors red; soon purple and bluish hues appear in the mixture, which are constant, for an hour; finally the sugar chars.

(3) Drop quietly 0.005 gm. ( $\frac{1}{40}$  gr.) of Gelsemin, without trying to dissolve it, into 5 c.c. (81 minims) of a very dilute mixture of a freshly prepared 10 per cent solution of potassium ferricyanid and 1 drop of Reagent 4; the alkaloid is soon covered with a precipitate of Berlin blue; within an hour a precipitate of Berlin blue settles from the whole fluid.

(4) Spread 5 drops of Reagent 22 out to a thin film, and drop a few separate particles of Gelsemin on it; a purple spot forms around every particle, soon disappearing.

### Test for Guaiacol, $C_6H_4 \cdot OH \cdot OCH_3$ .

Guaiacol is a colorless, clear, neutral, oily liquid, with an odor and taste suggestive of creosote; miscible with alcohol, ether, chloroform, caustic-soda solution, and carbon disulfid; slightly soluble in glycerin, liquid paraffin, and water; one part of Guaiacol dissolves in about two hundred parts of water at the normal temperature.

(1) Ferric-chlorid solution (Reagent 4) gives a brown turbidity in the *watery* solution of Guaiacol.

(2) A pure blue-colored fluid is obtained by the addition of a small quantity of ferric-chlorid solution (Reagent 4) to an *alcoholic* solution of Guaiacol; the blue color changes to green.

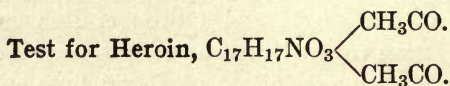
(3) Sulfuric acid (Reagent 21) dissolves Guaiacol with a yellow-red color, considerable heat being evolved in the process.

(4) A yellow fluid changing to a bright red is obtained by mixing 1 drop of Guaiacol, 5 drops of chloroform, and 10 drops of sulfuric acid (Reagent 21); the chloroform separates out colorless.

(5) One drop Guaiacol dissolves with a dark-brown color in 2 drops of Reagent 22.

(6) The least trace of Guaiacol dissolves in nitric acid (Reagent 15) with a bright-red color, soon changing to a dark brown-red; large quantities with a violent action.

(7) Guaiacol changes to a green and violet-colored fluid in contact with alkaline fluids and the atmosphere.



### *Diacetyl Morphin.*

Heroin is a white inodorous, crystalline powder, with a faintly bitter taste; insoluble in water, but soluble in dilute acids.

(1) If a few drops of nitric acid (Reagent 15) are added to a trace of Heroin, the latter is rapidly dissolved, producing a yellow solution which changes gradually (or rapidly if heated) to a greenish blue—the change apparently proceeding from the middle of the liquid. The color then gradually fades until the liquid has again acquired a bright-yellow color. This reaction is characteristic of Heroin.

### Test for Homatropin, $C_{16}H_{21}NO_3$ .

#### *Homatropin, Oxytoluylatropin.*

Homatropin is an artificial alkaloid prepared by evaporating a mixture of tropin and mandelic acid with diluted hydrochloric acid, crystallizing in very deliquescent prisms; soluble in ether, alcohol, and chloroform; less soluble in water and in carbon disulfid, alkaline to litmus paper when placed on it with a drop of water.

(1) Dissolve 0.002 gm. ( $\frac{1}{32}$  gr.) Homatropin in 1 drop nitric acid (Reagent 15); evaporate to dryness, and add an alcoholic potassium-hydrate solution (1:5) to the residue; a distinctly yellow color is produced.

(2) Dissolve 0.005 gm. ( $\frac{1}{12}$  gr.) of Homatropin, or one of its salts, in a little water; make alkaline with ammonia-water (Reagent 1); agitate in a separator with 10 c.c. (162 minims) of chloroform; collect the chloroform; evaporate. Now make a 2 per cent solution of mercuric chlorid in 50 per cent alcohol, and add 10 drops thereof to the residue from the evaporation of the chloroform; warm slightly; a brick-red precipitate is formed.

### Test for Hydrastin, $C_{21}H_{21}NO_6$ .

Hydrastin is in short, colorless, or somewhat opalescent, four-sided prisms; odorless and almost tasteless in consequence of its insolubility in the saliva; soluble in

chloroform, ether, and alcohol; nearly insoluble in water; the watery solution, however, has a very faintly bitter taste and is neutral to litmus paper.

(1) An aqueous solution of Hydrastin, or one of its salts (especially the acetate), has an acid reaction and very bitter taste, with a peculiar choking sensation; the solution of the acetate is greenish-blue fluorescent when not too dilute.

(2) Hydrastin thrown on sulfuric acid (Reagent 21) colors the latter yellow, changing to purple; nitric acid (Reagent 15) colors slowly yellow if present in very small quantities; the color is orange-red if the alkaloid is present in a large quantity; a green-blue fluorescent fluid is obtained with this acid test by diluting with water.

(3) The smallest particle of Hydrastin colors Reagent 22 bright red.

(4) A dark-green color reaction is obtained from a small particle of Hydrastin when strewn upon a mixture of 0.020 gm. ( $\frac{1}{3}$  gr.) ammonium molybdate and 5 drops sulfuric acid (Reagent 21), difference from morphin.

(5) Yellow to brown-yellow, red-yellow, and dark-brown color reactions are obtained if a small quantity of bismuth subnitrate is triturated to a soft mass with a few drops of sulfuric acid (Reagent 21) and a few particles of Hydrastin are strewn thereon.

### Test for Hyoscin, $C_{17}H_{23}NO_3$ .

Hyoscin is a brown, transparent, semi-liquid, balsam-like substance, soluble in alcohol, ether, and water.

(1) Hyoscin dissolved in nitric acid (Reagent 15) and dried on a water-bath is turned violet by an alcoholic solution of potassium hydrate.

(2) An aqueous solution of Hyoscin is precipitated by

potassium-hydrate solution but not by ammonia-water (Reagent 1).

(3) Hyoscin does not throw down mercuric oxide when added to a solution of mercuric chlorid and warmed on a water-bath (difference from atropin).

### Test for Hyoscyamin, $C_{17}H_{23}NO_3$ .

Hyoscyamin is in long, white, silky needles; has an alkaline reaction to phenolphthalein; an acrid, disagreeable taste; odorless; slightly soluble in water; very soluble in alcohol and ether; is quickly altered by contact with water and an alkali; when heated with potassa or soda is completely decomposed with disengagement of ammonia; neutralizes acids, and is precipitated by infusion of galls; dilates the pupil.

(1) An aqueous solution of Hyoscyamin acidulated with hydrochloric acid is not precipitated by platinic chlorid but is by gold chlorid, and this when dissolved in boiling water, acidulated with hydrochloric acid and crystallized, yields lustrous, golden-yellow scales (distinction from atropin).

### Test for Iodol, $C_4I_4NH$ .

#### *Tetraiodopyrrol.*

Iodol is a whitish or pale yellow crystalline powder; tasteless; odorless when strictly pure, but usually with a faint odor; becomes brown when heated on the water-bath and loses iodine; explodes when heated to  $140^{\circ} C.$ , giving off violet vapors and leaving a shining charcoal; very little soluble in boiling water (1:5000); easily soluble in ether, alcohol, ammonia-water (Reagent 1) on warming, and in carbon disulfid; less soluble in chloroform.

(1) A sediment of shining, black, heavy crystals is

formed if 0.050 gm. ( $\frac{3}{4}$  gr.) Iodol, 0.20 gm. (3 grs.) mercuric chlorid, and 5 c.c. (81 minims) of water are boiled together; they are formed, also, but more slowly, when 0.050 gm. ( $\frac{3}{4}$  gr.) Iodol is warmed for hours with 5 c.c. (81 minims) of Reagents 13 or 14.

(2) 0.020 gm. ( $\frac{1}{3}$  gr.) of Iodol strewn upon 2 c.c. (32 minims) sulfuric acid (Reagent 21) colors the latter yellow, green, and brown; a brighter and more constant green color is obtained when 2 drops of a ferric-chlorid solution (Reagent 4) is added to the acid.

(3) Boiling hydrochloric acid decomposes Iodol, and the solution colors brown giving off violet vapors of iodine.

(4) Iodol is decomposed by boiling nitric acid (Reagent 15) with a red color.

(5) A red fluid, changing to brown in a few minutes, is obtained if 0.020 gm. ( $\frac{1}{3}$  gr.) Iodol is agitated with 2 c.c. (32 minims) of Reagent 22.

(6) A dark-green residue with brown edges is obtained if 0.005 gm. ( $\frac{1}{12}$  gr.) Iodol is slowly evaporated in a flat-bottomed dish with 2 c.c. (32 minims) of water and 1 drop of ferric-chlorid solution (Reagent 4).

(7) Addition of 2 drops of a  $\frac{n}{10}$  silver solution to 0.020 gm. ( $\frac{1}{3}$  gr.) Iodol, dissolved in 10 c.c. (162 minims) of alcohol (0.83 sp. gr.) in the dark, reduces silver; the fluid becomes turbid, green to violet colored, and clears up slowly.

### **Test for Laudanin, $C_{20}H_{25}NO_4$ .**

Laudanin, an alkaloid of opium, forms small, rose-colored, tasteless granules, or rhombic crystals; gives a blue spot on moist red litmus paper; is easily soluble in chloroform and sodium-hydrate solution (Reagent 20); less soluble in alcohol, ether, or carbon disulfid.

(1) Colors chlorin-water (Reagent 3) yellow or faintly

red when agitated therewith, changing to a brown hue on the addition of ammonia-water (Reagent 1).

(2) At first Laudanin does not give any color reaction when strewn upon sulfuric acid (Reagent 21); a little later, however, a pink color turning to red is developed; a dark carmine-red color reaction is obtained when a drop of ferric-chlorid solution (Reagent 4, diluted 1:10) is added to the sulfuric acid (Reagent 21); the red color soon darkens into red-brown.

(3) Laudanin colors black, changing to a red-brown when a very small particle of sodium nitrate is added to the sulfuric acid (Reagent 21).

(4) Laudanin colors red-brown when strewn on nitric acid (Reagent 15).

(5) A permanent green-colored fluid is obtained when a few milligrams are agitated with 10 c.c. (162 minims) of a very dilute, nearly colorless ferric-chlorid solution (1 drop of Reagent 4 to 25 c.c. (405 minims) of water); neither chloroform nor carbon disulfid takes the color out.

(6) The watery solutions of Laudanin acetate are bitter.

#### **Test for Laudanosin, $C_{21}H_{27}NO_4$ .**

Laudanosin crystallizes in long needles; soluble in ether, alcohol, and chloroform; insoluble in water and in solution of the alkalies; gives a blue spot when strewn upon moist red litmus paper.

(1) Laudanosin colors chlorin-water (Reagent 3) yellow; ammonia-water (Reagent 1) develops a red color from the yellow.

(2) Laudanosin dissolves in sulfuric acid (Reagent 21) with a lilac color soon fading; touch the sulfuric acid with a glass rod moistened with ferric-chlorid solution (Reagent 4); the color changes to an intense violet.

(3) A bright red is obtained when a mixture of 0.005 gm.

( $\frac{1}{12}$  gr.) Laudanosin and 0.005 gm. ( $\frac{1}{12}$  gr.) sodium nitrite ( $\text{NaNO}_2$ ) is strewn upon a few drops of sulfuric acid (Reagent 21); a faintly pink color reaction is obtained when cane-sugar is substituted for the sodium nitrite; the color changes to brown-yellow.

(4) A yellow color reaction is obtained by treating Laudanosin with nitric acid (Reagent 15).

### Test for Menthol, $\text{C}_{10}\text{H}_{19}(\text{OH})$ .

Menthol crystallizes in long, colorless needles, with a peppermint odor and a warm, aromatic taste, followed by a sensation of cold when air is drawn into the mouth; it dissolves easily in ether, alcohol, chloroform, liquid paraffin, glacial acetic acid, and carbon disulfid; very slightly soluble in water, to which it imparts its odor and taste, and in caustic potash and soda solutions; applied to the skin it causes a peculiar sensation of cold; its alcoholic solution is neutral to litmus paper.

(1) A yellow liquid changing to a turbid brown-red color is obtained by shaking 0.50 gm. (8 grs.) Menthol with 20 c.c. (325 minims) sulfuric acid (Reagent 21).

(2) Menthol liquefies when triturated with chloral hydrate, camphor, phenol, resorcin, or thymol in the proportion of 2 parts of the former to 1 of the latter.

(3) Boiled with sulfuric acid (Reagent 21) diluted with half its volume of water, Menthol acquires an indigo-blue or ultramarine color, the acid becoming brown.

### Test for Methacetin, $\text{C}_6\text{H}_4 \cdot \text{OCH}_3 \cdot \text{NH}(\text{C}_2\text{H}_5\text{O})$ .

#### *Paraoxymethylacetanilid.*

Methacetin forms white, lustrous, scaly, odorless crystals or needles, with a taste suggestive of acetanilid; easily soluble in acetone, alcohol, and chloroform; less soluble

in ether and carbon disulfid; soluble in cold water (1:500) and in boiling water (1:12); soluble in glycerin and in olive-oil; neutral to litmus paper.

(1) Methacetin is easily soluble in sulfuric acid (Reagent 21), the solution being colorless at water-bath temperature, but of violet hue when heated higher.

(2) A red color is obtained when 0.010 gm. ( $\frac{1}{6}$  gr.) Methacetin is heated to boiling with 0.5 c.c. (81 minims) hydrochloric acid (Reagent 5), cooled, diluted with 3 c.c. (48 minims) water, and 1 drop of Reagent 17 added.

(3) If 0.10 gm. ( $1\frac{1}{2}$  gr.) Methacetin is warmed with 1 c.c. (16 minims) caustic-soda solution (Reagent 20), and 3 drops chloroform, it gives off the offensive poisonous odor of phenylisocyanid (the isonitril reaction).

(4) A bright green-colored fluid is obtained when a mixture of 0.030 gm. ( $\frac{1}{2}$  gr.) Methacetin and 0.030 gm. ( $\frac{1}{2}$  gr.) sodium nitrate ( $\text{NaNO}_3$ ) is strewn upon 1 c.c. (16 minims) sulfuric acid (Reagent 21); the fluid is violet colored when sodium nitrite ( $\text{NaNO}_2$ ) has been taken instead of the nitrate.

(5) Methacetin is soluble in nitric acid (Reagent 15) with a red-yellow color.

(6) 0.010 gm. ( $\frac{1}{6}$  gr.) Methacetin when shaken with 10 c.c. (162 minims) chlorin-water (Reagent 3) colors the latter red-yellow; a few drops ammonia-water (Reagent 1) changes this immediately to a brown-red.

### Test for Morphin, $\text{C}_{17}\text{H}_{19}\text{NO}_3 + \text{H}_2\text{O}$ .

Morphin crystallizes in white, shining, transparent, fine needles or prisms, or in a crystalline powder; very slightly soluble in water, ether, chloroform, benzene, and carbon disulfid; more soluble in alcohol and acetone; has an alkaline reaction to litmus paper; melts at  $200^\circ \text{C}$ ., forming a purple fluid, changing to a dark brown, later on to black.

(1) A blue fluid that can be diluted with water without fading is obtained when a few crystals of Morphin are moistened with water and 1 drop of Reagent 4 is added.

(2) Morphin gives no reaction with strictly pure sulfuric acid (Reagent 21) when strewn upon it in finely pulverized form, though in course of time the acid turns somewhat green; this mixture becomes dark purple or brown when touched with a pointed glass rod moistened with a faint trace of nitric acid (Reagent 15); more nitric acid changes the color to red.

(3) Crystals of Morphin become bright-red colored when strewn upon nitric acid (Reagent 15), while the acid itself becomes yellow-red; this color test of the acid is better observed when it has been previously diluted with twice its volume of water.

(4) Triturate 0.020 gm. ( $\frac{1}{5}$  gr.) ammonium molybdate with 5 drops sulfuric acid (Reagent 21), spread this out to a thin film and add a trace of Morphin, when the paste becomes permanently blue colored. Dissolve 0.010 gm. ( $\frac{1}{10}$  gr.) Morphin in 100 c.c. (3 f. oz.) water; dilute 10 c.c. (162 minims) of this solution to 1000 c.c. (34 f. oz.), and evaporate 10 c.c. (162 minims) of this to dryness, and add to it the freshly prepared mixture of ammonium molybdate and sulfuric acid; a lilac color appears immediately, soon changing to a permanent dark blue. Pink-violet and brown-red colors are obtained when titanio acid is substituted for the ammonium molybdate. Morphin gives a black or dark-brown color reaction if pure bismuth subnitrate is used instead of ammonium molybdate.

(5) Morphin dissolves in Reagent 22 with a faint green color, increasing in intensity when more potassium dichromate is added; the mixture becomes at last brown colored, but in course of time the green hue may come back.

(6) A rose-red mixture is obtained by triturating to-

gether 0.020 gm. ( $\frac{1}{3}$  gr.) Morphin with 0.010 gm. ( $\frac{1}{6}$  gr.) cane-sugar, and 2 drops sulfuric acid (Reagent 21).

(7) Morphin causes a blue precipitate in a mixture of ferric chlorid and potassium ferricyanid; the best way to observe it is to strew the Morphin, or its salt previously reduced to a fine powder, upon 10 c.c. (162 minims) of the mixture.

(8) A mixture of 0.10 gm. ( $1\frac{1}{2}$  grs.) potassium iodate ( $\text{KIO}_3$ ), 5 drops acetic acid, and 5 c.c. (81 minims) water may be warmed together on a water-bath, in a scrupulously clean test-tube, without decomposition; but iodine is liberated immediately upon the addition of 0.001 gm. ( $\frac{1}{64}$  gr.) of Morphin or one of its salts; chloroform extracts it with the well-known purple color.

(9) 0.050 gm. ( $\frac{3}{4}$  gr.) finely pulverized Morphin dissolves easily in 50 c.c. ( $1\frac{3}{4}$  f. oz.) of lime-water; chlorin-water (Reagent 3) colors this solution bright red.

(10) Strong chlorin-water is colored pale yellow when agitated with Morphin or its salts; a brown to red color is obtained at the place of contact when this mixture is carefully overlaid with ammonia-water (Reagent 1); the fluid becomes dark brown when the reagents mix.

(11) When 0.010 gm. ( $\frac{1}{6}$  gr.) Morphin is evaporated on a sand-bath with 10 c.c. (162 minims) of 10 per cent sulfuric acid, the solution dries at the edges with a gray, somewhat purplish tinged color; the fluid itself colors brown-red.

(12) A bright blue-colored fluid is obtained in the course of an hour, when 0.020 gm. ( $\frac{1}{3}$  gr.) Morphin, 0.020 gm. ( $\frac{1}{3}$  gr.) sodium nitrite ( $\text{NaNO}_2$ ), and 0.040 gm. ( $\frac{2}{3}$  gr.) mercuric chlorid are warmed and frequently stirred on the water-bath with 6 c.c. (97 minims) of water; the fluid afterwards becomes turbid, and a dark-blue crystalline precipitate settles down that turns brown in the course of a day; alcohol does not destroy the blue color. This

reaction may be hastened by warming the mixture over an open flame instead of on the water-bath; but the color is not so bright; a violet-blue is obtained when 0.020 gm. ( $\frac{1}{5}$  gr.) mercuric chlorid (instead of 0.040 gm.— $\frac{2}{5}$  gr.) and 4 c.c. (64 minims) instead of 6 c.c. (97 minims) of water are taken; with Morphin salts the reaction does not succeed as well as with the alkaloid; an amethyst color is obtained by employing mercuric bromid instead of mercuric chlorid (distinction from codein, narcein, narcotin, and papaverin).

(13) When added to a solution of iodic acid or an acidulous iodate, Morphin and its salts redden the liquid and set iodine free; various other organic substances act in a similar manner, but the color produced by these is removed by ammonia, while the redness produced by Morphin is greatly intensified by the addition of ammonia.

### Test for Naphthalin, $C_{10}H_8$ .

#### *Naphthalene.*

Naphthalin is in large white, thin crystalline scales, with a penetrating, disagreeable, coal-tar odor, and a burning, aromatic taste; it is soluble in ether, alcohol, chloroform, benzene, liquid paraffin, warm acetic acid, fixed and volatile oils, and carbon disulfid; insoluble in cold water, but when boiled with it imparts a faint odor and taste; is neutral to litmus paper moistened with alcohol.

(1) In the cold, concentrated sulfuric acid (Reagent 21) colors Naphthalin a little, if strictly pure, but if warmed on the water-bath gives a brown or red color reaction; yellow if nitric acid (Reagent 15) is substituted. The mixture becomes brown if the sulfuric acid contains a trace of nitric acid; the same color also develops slower when a trace of sodium nitrate ( $NaNO_3$ ) has been added to the sulfuric acid.

(3) A red, purple-tinged fluid is obtained when 0.10 gm. ( $\frac{1}{5}$  gr.) Naphthalin is triturated with a pointed glass rod previously moistened with a trace of ferric-chlorid solution (Reagent 4) and, drop by drop, 10 c.c. (162 minims) of sulfuric acid (Reagent 21) is added; the color changes to dark violet when warmed on the water-bath, and becomes red when diluted with water. Neither chloroform nor carbon disulfid extract the color from this mixture.

(3) Every particle of Naphthalin turns immediately black when strewn upon a mixture of 0.030 gm. ( $\frac{1}{2}$  gr.) sodium nitrite ( $\text{NaNO}_2$ ) and 5 c.c. (81 minims) sulfuric acid (Reagent 21).

(4) 2 c.c. (32 minims) sulfuric acid (Reagent 21) colors a bright red when slightly warmed and agitated with a solution of 0.050 gm. ( $\frac{3}{4}$  gr.) Naphthalin in 1 c.c. (16 minims) of chloroform; the chloroform remains colorless.

(5) Abundant yellow crystalline needles of Naphthalin picrate form if 0.090 gm. ( $1\frac{1}{2}$  grs.) Naphthalin and 0.160 gm. ( $2\frac{1}{2}$  grs.) picric acid are dissolved in 4 c.c. (64 minims) of warm alcohol (0.83 sp. gr.) and the solution allowed to cool.

### Test for Naphthol, $\text{C}_{10}\text{H}_7\text{OH}$ .

#### *Alpha- and Beta-Naphthol.*

Alpha-Naphthol crystallizes (from boiling water) in long, soft, colorless needles, soon coloring brown-red; is easily soluble in alcohol, ether, and benzene, very little so in cold water, with a faint odor recalling that of carboic acid, and a burning acrid taste.

(1) Shake a few crystals of Alpha-Naphthol with 50 c.c. ( $1\frac{3}{4}$  f. oz.) milk of lime; filter; add to the clear filtrate bromin-water (Reagent 2); a flocculent sediment of a lilac color, soon changing to violet, is produced.

(2) Mix 0.025 gm. (or  $\frac{1}{2}$  gr.) Alpha-Naphthol with

2.5 c.c. (40 minims) of 94 per cent alcohol, 7.5 c.c. (121 minims) water, and ten drops of ferric-chlorid solution (Reagent 4); a yellow-red flocculent sediment, soon changing to brown and afterwards to a lilac, forms in the mixture.

(3) 0.20 gm. (3 grs.) Alpha-Naphthol, 0.20 gm. (3 grs.) mercuric chlorid, and 0.10 gm. ( $1\frac{1}{2}$  grs.) of sodium nitrate ( $\text{NaNO}_3$ ) warmed on a water-bath with 10 c.c. (162 minims) of water give a bright-red precipitate.

Beta-Naphthol is in colorless, silky scales or yellowish-white crystalline powder, having a weak phenol-like odor and a sharp but not persistent taste; it is easily soluble in watery solutions of the alkalies, in ether, alcohol, benzene, chloroform, and oils; less soluble in carbon disulfid, with difficulty in liquid paraffin; soluble in boiling water 1:76, in water at  $15^\circ\text{C}$ . 1:1000; neutral to litmus paper moistened with alcohol.

(4) Beta-Naphthol colors cold sulfuric acid (Reagent 21) red-yellow; nitric acid (Reagent 15) colors red, whereby the crystals of Beta-Naphthol blacken; every particle of Beta-Naphthol that comes in contact with sulfuric acid containing a trace of nitric acid blackens immediately.

(5) Boil 1 gm. ( $15\frac{1}{2}$  grs.) Beta-Naphthol with 100 c.c. ( $3\frac{1}{2}$  f. oz.) of water, cool to  $15^\circ\text{C}$ .; filter; this filtrate colors green when a few drops of ferric-chlorid solution (Reagent 4) is added to it; it soon becomes turbid, and beautiful white crystals conglomerate in it; these crystals appear in a short time if 0.025 gm. (or  $\frac{1}{2}$  gr.) of Beta-Naphthol is dissolved in 25 c.c. (40 minims) of alcohol (sp. gr. 83), 75 c.c. ( $2\frac{1}{2}$  f. oz.) of water, and 10 drops of the ferric-chlorid solution are added; the clear fluid has a yellow-green color.

(6) Shake 50 c.c. ( $1\frac{3}{4}$  f. oz.) milk of lime with a few crystals of Beta-Naphthol and filter, whereby a highly

fluorescent solution is obtained, in which crystals of Beta-Naphthol form in large quantities after the addition of acetic or other acids. Bromin-water (Reagent 2) gives a dense turbidity, turning dirty green after the addition of ammonia-water (Reagent 1); a dark-brown flocculent sediment soon forms.

(7) Ammonia-water (Reagent 1) agitated with Beta-Naphthol becomes fluorescent.

(8) 0.10 gm. ( $\frac{1}{6}$  gr.) Beta-Naphthol and 0.160 gm. ( $\frac{1}{4}$  gr.) of picric acid dissolve when slightly warmed with 1 c.c. (16 minims) of alcohol of 94 per cent and 1.5 c.c. (24 minims) of water; on cooling they are deposited separately as yellow-red crystals.

(9) Beta-Naphthol is soluble in warm concentrated sulfuric acid with a green-violet color; it becomes red, soon changing to brown-black if it is strewn upon 5 c.c. (81 minims) of sulfuric acid containing 0.030 gm. ( $\frac{1}{2}$  gr.) sodium nitrite ( $\text{NaNO}_2$ ).

(10) A reaction for both Alpha- and Beta-Naphthol is to dissolve 0.010 gm. ( $\frac{1}{6}$  gr.) of the compound in 5 drops of chloroform; add 5 drops of a 10 per cent. caustic soda or potash solution; heat to  $50^\circ \text{C}$ .; the top layer turns blue; this blue layer sinks when ether is added; the whole fluid is blue if alcohol is added; it fades by addition of acetic acid.

(11) A piece of pine wood dipped into an aqueous solution of Beta-Naphthol and afterwards moistened with diluted hydrochloric acid becomes green on exposure to daylight.

### Test for Narcein, $\text{C}_{23}\text{H}_{29}\text{NO}_9 + 2\text{H}_2\text{O}$ .

Narcein crystallizes in long white prisms or needles; inodorous; having a bitter taste; soluble in alcohol, slightly so in carbon disulfid and chloroform, hardly soluble

in benzene and in ether; soluble in water at 100° C. 1:100, at 15° C. 1:425; easily soluble in lime-water; the solution becoming yellowish colored and changing to red after the addition of chlorin-water (Reagent 3); neutral to litmus paper.

(1) Narcein does not give the reaction with ferric-chlorid solution (Reagent 4) as morphin does; neither does it give the blue precipitate in the potassium ferri-cyanid mixture as morphin does.

(2) Narcein colors sulfuric acid (Reagent 21) brownish yellow; red and red-brown afterwards when strewn upon it; the mixture becomes brown-violet when stirred with a glass rod moistened with a trace of nitric acid (Reagent 15); a permanent cherry-red colored fluid is obtained when a solution of Narcein in diluted sulfuric acid (1.11 sp. gr.) is concentrated upon the water-bath.

(3) Narcein colors nitric acid (Reagent 15) red, which color soon fades to yellow; diluted nitric acid has no (or very little) effect upon it. Compare *Morphin*.

(4) Narcein gives a brownish-green changing to blue color reaction if treated with molybdate of ammonia and sulfuric acid, as described for *Morphin*; to bismuth subnitrate Narcein gives a similar reaction as morphin; the black color soon changing however to a lilac.

(5) Narcein gives a yellow-green mixture if triturated with sugar and sulfuric acid.

(6) Bromin-water (Reagent 2) gives an amorphous precipitate of a bright-yellow color in the watery solution of Narcein.

(7) Chlorin-water (Reagent 3) dissolves Narcein colorless; this solution becomes red colored when ammonia-water (Reagent 1) is added to it.

**Test for Narcotin,  $C_{22}H_{23}NO_7$ .**

Narcotin crystallizes in white, silky, flexible needles, usually larger than the crystals of morphin; inodorous and tasteless; has no effect on red litmus paper when strewn upon it and moistened afterwards with alcohol or water.

(1) A red color appears immediately if Narcotin is strewn upon slightly warmed sulfuric acid; also if stirred with a glass rod moistened with a trace of nitric acid (Reagent 15).

(2) Narcotin dissolves in nitric acid (Reagent 15) with a yellow color, changing to red in the course of an hour.

(3) A mixture of 0.020 gm. ( $\frac{1}{3}$  gr.) of Narcotin, 0.010 gm. ( $\frac{1}{6}$  gr.) of sugar, and 2 drops sulfuric acid (Reagent 21) triturated together colors faintly red; this color soon disappears.

(4) Narcotin colors red if treated with bismuth subnitrate, as described under *Morphin*.

**Test for Nicotin,  $C_{10}H_{14}NO_2$ .**

Nicotin, the alkaloid of tobacco, is a colorless or nearly colorless, oily liquid, having a strong, disagreeable odor, like that of an old pipe, and an exceedingly acrid, burning taste, even when largely diluted; it is inflammable; is entirely volatilizable, and, in the state of vapor, very irritating to the nostrils; is very soluble in water, alcohol, ether, the fixed oils, and oil of turpentine; strongly alkaline in its reaction toward litmus and phenolphthalein paper.

(1) Fifteen drops of Nicotin dissolved in 30 c.c. (1 f. oz.) of water remains clear when warmed; distinction from coniin solution.

(2) A glass rod moistened with hydrochloric acid (Re-

agent 5) gives off white clouds when brought near Nicotin or near a watery solution of it.

(3) Nicotin does not form crystals if treated as described for *Coniin* under (2).

(4) Mercuric-cyanid solution (Reagent 11) has no effect on a watery Nicotin solution; difference from *Codiin*.

(5) Tannin solution (Reagent 24) gives an amorphous precipitate in a watery solution of Nicotin.

(6) Perchlorid of platinum forms a yellowish-white precipitate in a watery solution of Nicotin; distinction from *Coniin*.

(7) Nicotin gives with nitric acid (Reagent 15) an orange color; with hydrochloric acid (Reagent 5) a violet color.

(8) Nicotin dissolved in ether and mixed with an ethereal solution of iodine yields an oily resin of brownish color; its presence may be detected by the familiar odor of stale tobacco.

**Test for Orexin Hydrochlorid,**  
 $\text{C}_6\text{H}_4 \cdot \text{CH}_2\text{N} \cdot \text{CH} \cdot \text{NC}_6\text{H}_5 \cdot \text{HCl} + 2\text{H}_2\text{O}.$

*Phenyldihydroquinazolin Hydrochlorid.*

Orexin Hydrochlorid, a derivative of quinolin, is a white crystalline powder; odorless; having a permanent bitter and burning taste; it violently irritates the nasal mucus membrane and induces sneezing; soluble in water at 15° C.; much less so in hot water; the watery solutions have an acid reaction to litmus paper; is easily soluble in alcohol; less so in chloroform; nearly insoluble in ether and carbon disulfid.

(1) Orexin Hydrochlorid is soluble without color in hydrochloric acid (Reagent 5), nitric acid (Reagent 15), sulfuric acid (Reagent 21), and sulfuric acid containing

chromic acid (Reagent 22); it colors brown if warmed with Reagent 21.

(2) A brown color reaction fading to green takes place when a mixture of 0.020 gm. ( $\frac{1}{3}$  gr.) Orexin Hydrochlorid and 0.020 gm. ( $\frac{1}{3}$  gr.) sodium nitrite ( $\text{NaNO}_2$ ) is strewn upon 1 c.c. (16 minims) sulfuric acid (Reagent 21); the color is faintly red-yellow when sodium *nitrate* ( $\text{NaNO}_3$ ) is substituted for the nitrite.

(3) A strong sickening odor of carbylamin is developed when equal parts of Orexin Hydrochlorid and zinc-dust are heated over the flame in a glass tube (isonitril reaction). Bleaching-powder ( $\text{CaOCl}_2$ ) solution gives a blue color to the filtered liquid when the residue has been treated with diluted (1:1) hydrochloric acid (anilin reaction).

(4) Orexin Hydrochlorid leaves no residue when ignited on platinum foil.

### Test for Papaverin, $\text{C}_{20}\text{H}_{21}\text{NO}_4$ .

Papaverin, an alkaloid of opium, is a crystalline powder; tasteless; easily soluble in acetone, alcohol, and chloroform; less soluble in ether and carbon disulfid; almost insoluble in water even by long boiling; neutral to litmus paper.

(1) Papaverin colors lilac when strewn upon sulfuric acid (Reagent 21); this color is more brown-red if a particle of sodium nitrate ( $\text{NaNO}_3$ ) or nitric acid (Reagent 15) has been added to the sulfuric acid.

(2) Sulfuric acid (Reagent 21) gives a brown-black color reaction, afterwards turning red when a mixture of equal parts of sodium nitrite ( $\text{NaNO}_2$ ) and Papaverin are strewn upon it.

(3) Papaverin colors Reagent 22 brown slowly.

(4) Papaverin turns dark brown to bluish black when chlorin-water (Reagent 3) is poured over it. It sinks in

yellow-brown streaks to the bottom when it is strewn upon chlorin-water (Reagent 3).

(5) Papaverin gives a brown color reaction if treated with bismuth subnitrate, as described under article *Morphin*; molybdic, titanio, and tungstic acids give lilac-colored reactions.

(6) 0.010 gm. ( $\frac{1}{10}$  gr.) Papaverin dissolves colorless in 1 c.c. (16 minims) of nitric acid (Reagent 15); this solution colors yellow in course of time—sooner by warming.

### Test for Paracotoin, $C_{19}H_{12}O_6$ .

Paracotoin, the chief active principle of *Paracoto bark*, is a faint yellow crystalline powder having no taste; very soluble in chloroform; less soluble in ether, alcohol, and carbon disulfid; almost insoluble in water.

(1) A faintly yellow neutral fluid is obtained when 0.10 gm. ( $\frac{1}{10}$  gr.) of Paracotoin is boiled for a while with 100 c.c. (3.38 f. oz.) of water; this solution is hardly bitter, and ferric-chlorid solution (Reagent 4) does not give any color to it (distinction from cotoin).

(2) Paracotoin gives a greenish-brown color reaction with sulfuric acid (Reagent 21), which darkens when sodium nitrite ( $NaNO_2$ ) has been added.

(3) Paracotoin is soluble in nitric acid (Reagent 15) with a permanent bright-yellow color.

### Test for Paraldehyd, $C_6H_{12}O_3$ .

Paraldehyd is a colorless, transparent liquid, having a strong, somewhat ethereal, but not unpleasant or pungent odor and a burning and cooling taste. Soluble in 8.5 parts of water at  $15^\circ C$ . and in 16.5 parts of boiling water; soluble in all proportions in alcohol, ether, chloroform, carbon disulfid, liquid paraffin, benzene, and fixed or

volatile oils. It has a very slight acid reaction to litmus paper. When cooled to nearly  $0^{\circ}\text{C}$ . it solidifies to a crystalline mass, which becomes liquid again at  $10.5^{\circ}\text{C}$ . It boils at  $123^{\circ}$ – $125^{\circ}\text{C}$ ., giving off inflammable vapors. It is converted into aldehyd when distilled with sulfuric acid, to be recognized by its irritating odor and by its property of reducing silver from a silver-nitrate solution.

(1) A mixture of 1 c.c. of sulfuric acid (Reagent 21) and 5 drops Paraldehyd soon becomes brown colored.

(2) A greenish-yellow color reaction soon changing to brown and black takes place when 1 drop of Paraldehyd is carefully spread out on a porcelain lid and a few drops of sulfuric acid (Reagent 21) added; a violent action takes place when larger quantities of Paraldehyd and sulfuric acid are added together; the mixture chars.

(3) Caustic soda has no action upon Paraldehyd; it remains.

(4) Paraldehyd does not immediately reduce silver from a solution of silver nitrate, neither in the cold nor by warming—only after a while.

### Test for Pelletierin Tannate.

Pelletierin tannate is an amorphous, pale, yellowish-gray powder having an astringent taste; soluble in warm dilute acids and in alcohol; hardly soluble in water; has a faint acid reaction to litmus paper.

(1) Lime-water gives a greenish-blue precipitate; ferric-chlorid solution (Reagent 4) gives a bluish-black precipitate in the filtrate of a mixture of 0.010 gm. ( $\frac{1}{10}$  gr.) of the preparation with 5 c.c. (81 minims) of water.

(2) Red litmus paper does not become blue when it is kept near the surface of a mixture of 1 gm. ( $15\frac{1}{2}$  grs.) of the preparation 0.050 gm. ( $\frac{3}{4}$  gr.) slaked lime and 10 c.c. (162 minims) of water.

(3) Chloroform (or ether) leaves on evaporation a few colorless drops of an alkaline reaction when agitated with the mixture described under (2). These drops have a peculiar odor and are nearly tasteless. Pelletierin is a liquid alkaloid of a somewhat aromatic odor obtained from pomegranate bark.

(4) The alkaline drops (3) give white clouds from a glass rod moistened with hydrochloric acid (Reagent 5).

The most valuable identity reaction, however, is its applicability as a tænicide (against tapeworm).

### Test for Phenacetin, $(C_6H_4(NH \cdot CH_3CO)O \cdot C_2H_5)$ .

#### *Para-acetphenitidin.*

Phenacetin occurs in small crystalline scales which are colorless, tasteless, and odorless; soluble in alcohol, 1:16; in ether, 0.5:100; in chloroform, 3.3:100; much less in carbon disulfid; in water at 100° C., 1:70, at 20° C., 1:2000. Ignited on platinum foil it leaves no residue. Shining crystals form when the warm saturated solution of Phenacetin is allowed to cool. Its watery solution does not affect litmus paper.

(1) Phenacetin dissolves colorless in sulfuric acid (Reagent 21) even when warmed with it on the water-bath and a trace of ferric-chlorid solution (Reagent 4) is added. In warm sulfuric acid it dissolves often with a blue color; this blue color may be due to an impure acid.

(2) Cold nitric acid (Reagent 15) dissolves Phenacetin with an orange color, difference from acetanilid and antipyrin, on which cold nitric acid has no effect.

(3) 0.10 gm. ( $1\frac{1}{2}$  grs.) Phenacetin is dissolved in 2 c.c. (32 minims) of hydrochloric acid (Reagent 5) only by warming; this solution becomes turbid on addition of 8 c.c. (130 minims) of water, but clears up again on warm-

ing. It becomes purple colored on addition of 1 drop of potassium dichromate solution (Reagent 17); bright red on addition of 0.5 c.c. (8 minims) of ferric-chlorid solution (Reagent 4),—this color soon changes, however, to a dirty green.

(4) 0.50 gm. (8 grs.) Phenacetin dissolves when added to 2 gms. (31 grs.) chloral hydrate previously melted on the water-bath ( $100^{\circ}$ ); and the solution is colorless if the Phenacetin is free from paraphenitidin; it is intensely violet colored if the paraphenitidin impurity is present. In the course of ten hours or less the solution becomes slightly rose colored.

(5) A dark-violet colored solution which turns green in a few hours is obtained if 0.030 gm. ( $\frac{1}{2}$  gr.) Phenacetin is triturated with an equal part of sodium nitrate ( $\text{NaNO}_2$ ) and this mixture strewn upon 1 c.c. (16 minims) sulfuric acid (Reagent 21). The color reaction is immediately green when sodium *nitrate* ( $\text{NaNO}_3$ ) is substituted for the nitrite; the green color soon turns brown.

(6) A pleasant fruit-like odor of ethyl acetate is liberated by warming from a mixture of 0.50 gm. (8 grs.) Phenacetin 1 c.c. (16 minims) alcohol and 2 c.c. (32 minims) sulfuric acid (Reagent 21).

(7) The offensive, poisonous odor of phenylisocyanid is liberated when 0.20 gm. (3 grs.) Phenacetin is boiled for a while with 5 c.c. (81 minims) of a 10 per cent caustic-soda solution, the fluid allowed to cool, 0.020 gm. ( $\frac{1}{3}$  gr.) chloral hydrate added, and the mixture warmed again.

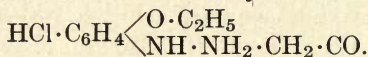
(8) A brown-yellow paste of crystalline nitrophenacetin is obtained when 0.20 gm. (3 grs.) Phenacetin is triturated with 2 c.c. (32 minims) nitric acid (Reagent 15). The crystals dissolve in 50 c.c. ( $1\frac{1}{2}$  f. oz.) of water at  $100^{\circ}$  C., forming a yellow-red solution, and bright-yellow crystal needles form if the solution is allowed to cool; they dis-

solve in an alcoholic caustic-soda solution with intense red color.

(9) A red-brown colored fluid is obtained in the course of half an hour when 5 drops of ammonia-water (Reagent 1) are added to 10 c.c. (162 minims) of a saturated watery solution of Phenacetin previously diluted with an equal volume of chlorin-water (Reagent 3).

(10) A solution of Phenacetin in hot water is colored violet and red by chlorin-water (Reagent 3).

### Test for Phenocoll Hydrochlorid,



### *Amidoacetparaphenitidin Hydrochlorid. Glycocollparaphenitidin.*

Phenocoll Hydrochlorid is a white crystalline powder of a salty with a sweet after-taste, possessing an aromatic odor; soluble 1:16 in water at normal temperature, in much less at 100°; these solutions have no reaction upon litmus paper; colorless, short prisms form in the hot saturated watery solution when allowed to cool; very soluble in alcohol; scarcely soluble in ether, chloroform, and carbon disulfid.

(1) Phenocoll Hydrochlorid chars if heated to about 200°, when salicylic acid can be detected in the residue by its action with ferric-chlorid solution (Reagent 4).

(2) Potassium ferrocyanid solution (Reagent 19) gives a pale-yellow precipitate soon crystallizing in a saturated aqueous solution of Phenocoll Hydrochlorid.

(3) Strewn upon sulfuric acid (Reagent 21) Phenocoll Hydrochlorid gives a pale-yellow color reaction which becomes darker in time; addition of sodium nitrate ( $\text{NaNO}_3$ ) or nitrite ( $\text{NaNO}_2$ ) changes it to dark red-brown.

(4) With nitric acid (Reagent 15) Phenocoll Hydrochlorid gives a permanent greenish-yellow color reaction; a bright-red clear solution is obtained if 0.50 gm. (8 grs.) of Phenocoll Hydrochlorid is heated to boiling with 1 c.c. (16 minims) of the acid.

(5) Large yellow crystals form if 1 c.c. (16 minims) of the saturated watery solution of Phenocoll Hydrochlorid is diluted with 9 c.c. (146 minims) of water and 1 c.c. (16 minims) of picric-acid solution (Reagent 16) is added.

### Test for Picrotoxin, $C_{30}H_{34}O_{13}$ .

Picrotoxin is a bitter, white, crystalline, odorless substance obtained from *Cocculus indicus*; soluble in water  $15^\circ$ , 1:400, the solution being intensely bitter, with no reaction on litmus paper; caustic potash, soda, or ammonia solutions of 10 per cent dissolve it easier; also soluble in glacial acetic acid and in alcohol; less soluble in ether or chloroform.

(1) Picrotoxin dissolves in sulfuric acid (Reagent 21) to a yellow-brown fluorescent fluid (changing to red-brown in the course of a day), in which water does not cause turbidity as in a phosphoric-acid solution.

(2) A green-yellow color reaction is obtained when equal parts of Picrotoxin and caustic soda are moistened with a drop of water; the green color changes to red-brown.

(3) A bright-red color reaction is obtained with Picrotoxin as follows: Add 1 drop sulfuric acid (Reagent 21) to 0.005 gm. ( $\frac{1}{12}$  gr.) sodium nitrate ( $NaNO_3$ ); spread out on a porcelain lid; strew a mixture of 0.050 gm. ( $\frac{3}{4}$  gr.) Picrotoxin and 0.050 gm. ( $\frac{3}{4}$  gr.) sodium hydrate ( $NaHO$ ) in the midst thereof; stir with a glass rod moistened with alcohol or water.

(4) Picrotoxin (0.020 gm. ( $\frac{1}{3}$  gr.)) reduces in half an

hour in the cold—quicker by warming—a diluted cupric-tartrate solution (0.5 c.c. (8 minims) to 2.5 c.c. (40 minims) cold water). Reduces also an ammoniacal  $\frac{n}{10}$  silver solution.

(5) A mixture of equal parts sugar and Picrotoxin is colored red by the addition of sulfuric acid (Reagent 21).

### Test for Pilocarpin, $C_{11}H_{16}N_2O_2$ .

Pilocarpin is an alkaloid found in jaborandi; the official salt is the hydrochlorate, and is in small, white crystals, odorless, and having a faintly bitter taste; deliquescent on exposure to damp air; very soluble in water and in alcohol; almost insoluble in ether or chloroform. The aqueous solution (1 in 100) of the salt is neutral or has a faintly acid reaction upon litmus paper.

(1) A characteristic reaction, had in common with cocain, is that Pilocarpin reduces calomel to Hg (blackens it) when 0.010 gm. ( $\frac{1}{10}$  gr.) of the alkaloid or the hydrochlorate and 1 gm. ( $15\frac{1}{2}$  grs.) of mercurous chlorid are triturated together and moistened by the breath.

(2) Another reaction is the behavior of the precipitate obtained from Reagent 12 in a diluted solution of the hydrochlorate; the amorphous precipitate sinks to the bottom, in the course of a day, in oily drops.

(3) Reagent 7 gives in Pilocarpin solutions a brown precipitate that often crystallizes to feathery brown crystals of serrated form, something like the blade of a scroll-saw.

(4) If one volume of Reagent 7 is mixed with two volumes of Pilocarpin solution (1:50), feathery black crystals are formed in abundance.

(5) Mix a few drops of Pilocarpin solution with a drop of sulfuric acid (Reagent 21); then let another drop of

sulfuric acid fall near it. Into the second drop put a fragment of potassium dichromate ( $K_2Cr_2O_7$ ), let it digest a moment and then stir the drops together, when a vivid emerald-green is obtained.

**Test for Piperazin,**  $NH\langle\begin{smallmatrix} CH_2 \cdot CH_2 \\ CH_2 \cdot CH_2 \end{smallmatrix}\rangle NH$ .

*Diethylendiamin. Hexahydropyrazin.*

Piperazin forms a crystalline mass of colorless prismatic crystals; of a faint, peculiar odor, and a taste suggestive of saltpetre and ammonium chlorid; is very hygroscopic, and attracts carbonic acid from the atmosphere, whereby it liquefies to a carbonate; has an alkaline reaction to litmus paper and phenolphthalein; is easily soluble in water and in alcohol; less soluble in ether; is soluble in lime-water, without liberating ammonia. The watery solution decomposes calomel.

(1) Hydrochloric acid (Reagent 5) gives off dense white clouds when a glass rod moistened with a 10 per cent Piperazin solution is held close to it.

(2) A saturated solution of picric acid (Reagent 16) gives a crystalline precipitate in a watery solution of (1:200) Piperazin.

(3) Sulfur is liberated from carbon disulfid when Piperazin is triturated therewith.

**Test for Piperin,**  $C_{17}H_{19}NO_3$ .

Piperin is in colorless or pale yellowish, shining, prismatic crystals; odorless and almost tasteless when first put in the mouth, but on prolonged contact producing a sharp and biting sensation; permanent in the air; almost insoluble in water; soluble in alcohol, ether, chloroform, acetone, benzene, and carbon disulfid; very soluble in hot acetic acid, giving a yellow-colored fluid

from which it separates into drops when allowed to cool. The alcoholic solution is neutral to litmus paper.

(1) Piperin 0.001 gm. ( $\frac{1}{84}$  gr.) dissolves in 5 drops of sulfuric acid (Reagent 21) with a yellow color, changing immediately to a red; on being breathed upon it fades to a greenish yellow.

(2) Piperin colors yellow-red when strewn upon nitric acid (Reagent 15); the acid itself colors yellow.

(3) The addition of a trace of sodium nitrite ( $\text{NaNO}_2$ ) to its acetic-acid solution gives a red color reaction.

(4) Piperin acts with bismuth subnitrate like morphin, with the difference, however, that the black color is preceded for a moment by a yellow-red color.

### Test for Pyridin, $\text{C}_5\text{H}_5\text{N}$ .

Pyridin is a colorless, very mobile fluid, permanent in the light; of a strong, persistent, empyreumatic odor, readily detected in large dilutions; a burning aromatic, not permanent, bitter taste; is highly inflammable and burns with a sooty flame; dissolves easily in water, which solution has a strongly alkaline reaction to litmus paper; miscible with alcohol, ether, liquid paraffin, benzene, chloroform, and fatty oils.

(1) Hydrochloric acid (Reagent 5) gives white clouds when brought near to a solution of Pyridin.

(2) Pyridin gives a brown turbidity in iodine-water (Reagent 6) which soon clears up; small brown, crystalline needles form in the fluid, by which the latter is decolorized.

(3) Reagent 7 solidifies, upon the addition of Pyridin, to a crystalline magma.

(4) Solution of tannin (Reagent 24) gives a heavy precipitate in a 2 per cent watery Pyridin solution.

(5) Calomel blackens when agitated with Pyridin.

**Test for Pyrogallol,  $C_6H_3(OH)_3$ .***Pyrogallic Acid.*

Pyrogallol is a bulky, white crystalline powder (or thin plates); odorless and having a bitter taste; soluble in water, alcohol, and ether; very soluble in boiling water and in boiling alcohol; very little soluble in chloroform, benzene, or carbon disulfid. The watery solution has an acid reaction to litmus paper.

(1) Soluble in sulfuric acid (Reagent 21) with a canary-yellow color, which soon disappears.

(2) Soluble in nitric acid (Reagent 15) with violent action and an olive-greenish-brown color.

(3) A freshly prepared solution of Pyrogallol gives a brown or black precipitate in a watery copper sulfate solution (1:14).

(4) A clear crystal of ferrous sulfate causes in the freshly prepared, colorless, watery solution of Pyrogallol (1:10) an indigo-blue color; this color increases in a short time, oxygen being absorbed; is more violet when a trace of sodium acetate or calcium carbonate has been added to the mixture.

(5) The solution (3) becomes red on addition of ferric-chlorid solution (Reagent 4), and the ferric chlorid is thereby reduced to ferrous chlorid.

(6) 2 c.c. (32 minims) of Pyrogallol solution and 1 c.c. (16 minims) of ammonia-water (Reagent 1) give a bright-yellow-colored fluid, soon turning brown.

(7) 0.010 gm. ( $\frac{1}{6}$  gr.) Pyrogallol dissolves in 10 c.c. (162 minims) lime-water (Reagent 10) with a lilac color; the solution soon becomes turbid and turns brown or black.

(8) Borax tinges Pyrogallol solution slowly red; sodium acetate colors it yellow in the course of a day.

(9) A solution of Pyrogallol reduces silver immediately when a  $\frac{n}{10}$  silver-nitrate solution is dropped into it.

**Test for Quinidin Sulfate,  $(C_{20}H_{24}N_2O_2)_2SO_4H_2 + 2H_2O$ .**

Quinidin Sulfate is in long, white, needle-like crystals, odorless, and having a very bitter taste; soluble in water, 1:100, at 15°; easily in 94 per cent alcohol, and in chloroform; almost insoluble in ether; its solution is neutral or faintly alkaline to litmus paper, and when treated with sulfuric acid has a decided blue fluorescence.

(1) A 10 per cent potassium-iodid solution gives a white precipitate in the watery saturated solution of Quinidin Sulfate (difference from quinin sulfate).

(2) Quinidin Sulfate gives the herapathit reaction when treated as described under quinin sulfate; the crystals are more *red* than from quinin—not green or brownish green.

(3) On treating 10 c.c. (162 minims) of an aqueous solution (1:1600) of Quinidin Sulfate with 2 drops of bromin-water (Reagent 2), and then with an excess of ammonia-water (Reagent 1) the liquid will acquire an emerald-green color.

**Test for Quinin Sulfate,  $(C_{20}H_{24}N_2O_2)_2SO_4H_2 + 7H_2O$ .**

*Neutral Quininsulfate. Diquininsulfate.*

Quinin Sulfate occurs in white, silky, light and fine, needle-shaped crystals; odorless, and having a persistent, very bitter taste; melts with a yellow color in its own water of crystallization when carefully heated in a glass tube over the open flame. It soon becomes red colored, however; a carmine-red solution is obtained if the melted mass is allowed to cool and warmed with alcohol. It

is soluble (1:800) in water at 15°; in glycerol and in alcohol; freely soluble in dilute acids; the aqueous solution is neutral to litmus paper, and has, especially when acidulated with sulfuric acid, a vivid, blue fluorescence.

(1) Quinin Sulfate dissolves in nitric or sulfuric acid, even when warm, either colorless or only with a very slightly yellow color. (Difference from morphin.)

(2) 1 c.c. (16 minims) of the cold, saturated, watery solution of Quinin Sulfate gives the thalleioquin reaction by mixing with 1 c.c. (16 minims) of chlorin-water (Reagent 3), and then slowly adding a few drops of ammonia-water (Reagent 1); the color is sea-green in this instance.

(3) A bluish-green thalleioquin reaction is obtained from the addition of 1 c.c. (16 minims) of ammonia-water (Reagent 1) to a mixture of 1 c.c. (16 minims) of a watery solution of Quinin Sulfate, 1 c.c. (16 minims) alcohol of 94 per cent, and 1 drop of bromin-water (Reagent 2), the latter to be taken with a glass rod. *No reaction is obtained when the substances are not mixed in the order named*, that is to say, in the Quinin solution, alcohol and ammonia-water are mixed first and the drop of bromin-water added afterwards. 15 to 20 drops of 10 per cent diluted sulfuric acid changes the bluish-green color to red.

(4) 0.5 c.c. (8 minims) of a 5 per cent potassium ferrocyanid solution produces a carmine-red color in 1 c.c. (16 minims) of the watery saturated solution of the Quinin Sulfate to which, on a glass rod, 1 drop of strong bromin-water (Reagent 2) has been added.

(5) A distinctive characteristic test for Quinin is the insolubility in cold alcohol, ether, chloroform, or water, and the very slight solubility in boiling alcohol of the microcrystalline precipitate of herapathit obtained in the following reaction: Dissolve 0.50 gm. (8 grs.) of Quinin Sulfate in 15 c.c. (243 minims) alcohol of 0.83 sp. gr. diluted with 5 c.c. (81 minims) of water, and acidulate the

fluid with 2 c.c. (32 minims) of 10 per cent sulfuric acid; add to this solution a solution 0.20 gm. (3 grs.) iodine in 10 c.c. (162 minims) alcohol of 0.83 sp. gr.; warm the mixture slightly and allow to cool. Hereby forms a crystalline precipitate of dark-green crystals by transmitted light, and shining with a metallic lustre when observed by reflected light.

(6) A 10 per cent potassium iodide (KI) solution does not give a precipitate in the cold, saturated, watery solution of Quinine Sulfate (difference from quinidine sulfate).

### Test for Quinoline, $C_9H_7N$ .

Quinoline is a colorless, oily liquid, turning reddish brown on exposure to the air; having an aromatic odor, resembling that of oil of bitter almonds; when heated to the boiling-point its vapors are inflammable; very little soluble in water, which solution has a pungent, hot taste, suggestive of oil of peppermint and has no reaction on litmus paper; miscible in all proportions with alcohol, ether, and chloroform; it easily dissolves camphor and resin.

(1) Quinoline partly reduces calomel—colors it gray.

(2) Reagents 2, 6, 7, and 12 form white crystalline precipitates in a watery solution of Quinoline, which are colored yellow by hydrochloric acid (Reagent 5).

### Test for Resorcin, $C_6H_4(OH)_2$ .

#### *Metadihydrobenzol. Resorcinol.*

Resorcin is prepared by fusing sodium benzene disulfonate with caustic soda, and forms colorless prisms or needles, turning yellow-brown or reddish pink when exposed to light and air; has an odor suggestive of a benzoic salt and urine; soluble in water, which solution has a disagreeable, sweetish, and afterwards pungent taste;

also easily soluble in alcohol and in ether or glycerin; hardly soluble in benzene, chloroform, or carbon disulfid; reddens moist blue litmus paper.

(1) Resorcin dissolves in lime-water (Reagent 10) with a faint lilac color, which changes to a yellow-green. It dissolves in a caustic-soda solution with a lilac color, which turns green or red if chloral hydrate or chloroform is added, which latter color brightens by warming to  $50^{\circ}$ ; it fades on the addition of acids.

(2) When dissolved in chlorin-water (Reagent 3) it colors the latter brown—very temporarily lilac; the solution turns bright red when ammonia-water (Reagent 1) is added.

(3) Resorcin colors bromin-water (Reagent 2) lilac—this color disappears immediately; the solution discolors, and a flocculent precipitate is thrown down.

(4) Resorcin is soluble in nitric acid (Reagent 15) with a red color, soon turning brown.

(5) Resorcin is soluble in sulfuric acid (Reagent 21) with a yellow color; a violet color reaction is obtained with sulfuric acid when a trace of nitric acid (Reagent 15) has previously been added to it. A trace of sodium nitrate ( $\text{NaNO}_3$ ) added to the sulfuric acid (Reagent 21) gives a greenish color reaction, turning brown and violet. Resorcin gives a bright-blue color reaction if strewn upon sulfuric acid (Reagent 21) in which sodium nitrite ( $\text{NaNO}_2$ ) has been dissolved.

(6) A dark-violet color reaction is obtained by adding a drop of ferric-chlorid solution (Reagent 4) to a watery solution of Resorcin.

(7) An intense carmine-red fluid is obtained by warming carefully over the flame a mixture of 0.050 gm. ( $\frac{3}{4}$  gr.) Resorcin with 0.10 gm. ( $1\frac{1}{2}$  grs.) tartaric acid and 10 drops sulfuric acid (Reagent 21); the mixture becomes blue when oxalic acid is substituted for the tartaric;

the color of the blue compound changes to an orange-red, which fades by diluting with 10 c.c. (162 minims) of water. The fluid shows dichroism by saturating with strong ammonia-water, is red by transparent, green by reflected, light. One obtains a red fluid with bluish-green fluorescent properties if citric instead of oxalic or tartaric acid has been used. Succinic acid, taken instead of citric, oxalic, or tartaric, produces a green fluorescence.

(8) Resorcin reduces silver from a  $\frac{n}{10}$  silver solution made alkaline by ammonia-water (Reagent 1).

**Test for Saccharin,  $C_6H_4\langle\begin{smallmatrix} CO \\ SO_2 \end{smallmatrix}\rangle NH$ .**

*Glusidum. Benzoyl-sulphonicimide.*

Saccharin is a sweet imide derivable from the toluene of coal-tar, and occurs as a bulky, white powder with slight aromatic odor suggestive of oil of bitter almonds, which becomes stronger on warming, and a remarkably sweet taste, being nearly 300 times as sweet as cane-sugar. It is soluble in 25 to 30 parts of boiling water, from which solution it crystallizes when cold; about 1:300 part of the Saccharin remains in solution. This saturated solution has an acid reaction to blue litmus paper; it is soluble in alcohol; less soluble in ether; much less in chloroform, benzene, and carbon disulfid; is easily soluble in ammonia-water (Reagent 1) and in caustic-potash and soda solutions, from which it is precipitated by the addition of acids.

(1) Saccharin dissolves colorless in nitric or sulfuric acid (Reagents 15 and 21) even by warming on the water-bath.

(2) A similar fluorescent reaction, as described under (7),

article *Resorcin*, is obtained as an identity reaction of Saccharin as follows:

0.005 gm. ( $\frac{1}{2}$  gr.) Saccharin, 0.005 gm. ( $\frac{1}{2}$  gr.) resorcin, and 3 drops of sulfuric acid (Reagent 21) are heated in a test-tube over the flame until white vapors appear—color reactions from yellow to red and afterwards olive-green are seen; cool, dilute with 10 c.c. (162 minims) of water; the fluid now has dichroistic properties; is yellow by transmitted light; green by reflected light, which becomes very prominent when saturated with 10 per cent NaOH solution, so that it is pink by transmitted and brilliant green by reflected light.

(3) 1 c.c. (16 minims) of a freshly prepared 5 per cent potassium ferrieyanid solution added to 10 c.c. (162 minims) of a saturated watery Saccharin solution and boiled gives a dark grass-green-colored fluid.

(4) A mixture of 0.10 gm. ( $1\frac{1}{2}$  grs.) Saccharin and 0.50 gm. (8 grs.) calcium carbonate heated in a glass tube gives off acid vapors and an odor of bitter almond oil; ammonia is liberated if slacked lime is substituted for the calcium carbonate.

(5) Phenol is produced by heating Saccharin with slaked lime; drops of phenol appear when 0.10 gm. ( $1\frac{1}{2}$  grs.) Saccharin is heated with 0.50 gm. (8 grs.) of powdered slaked lime. Transfer these drops of phenol with a glass rod into 1 c.c. (16 minims) of sulfuric acid, to which 0.001 gm. ( $\frac{1}{84}$  gr.) of potassium chlorate ( $\text{ClO}_3\text{K}$ ) has been previously added; add 1 c.c. (16 minims) of chloroform and shake the mixture; the under layer will have a green color.

(6) On fusing Saccharin with caustic soda ( $\text{NaHO}$ ), cooling, dissolving in water, faintly acidulating with hydrochloric acid (Reagent 5), and adding ferric chlorid ( $\text{Fe}_2\text{Cl}_6$ ) a reddish-purple color is obtained.

**Test for Salicin,  $C_{13}H_{18}O_7$ .**

Salicin is a crystalline glucoside obtained from the willow and the poplar. It is in colorless or white, silky, shining crystalline needles or a crystalline powder, odorless, and having a very bitter taste; soluble in water, alcohol, glacial acetic acid, and in caustic-potash and soda solutions; very sparingly soluble in ether; insoluble in chloroform and carbon disulfid; is neutral to litmus paper.

(1) A particle of Salicin strewn upon a few drops of sulfuric acid (Reagent 21) colors blood-red immediately; the acid remains colorless; slowly the Salicin dissolves in the acid and colors this red.

(2) Salicin is soluble in an alkaline copper-tartrate solution (Fehling's fluid), but decomposes it in a few hours, and separates red cuprous oxid when warmed on the water-bath; this decomposition appears immediately at 100°.

(3) A few particles of Salicin strewn upon a freshly prepared mixture of 0.020 gm. ( $\frac{1}{3}$  gr.) ammonium molybdate in 5 drops sulfuric acid (Reagent 21) become purple-colored immediately.

(4) The aromatic odor of salicylaldehyd is developed by warming 0.050 gm. ( $\frac{1}{3}$  gr.) Salicin, dissolved in 2 c.c. (32 minims) of potassium dichromate solution (Reagent 17), with 5 drops sulfuric acid (Reagent 21), until it commences to color brown.

**Test for Salicylic Acid,  $C_6H_4(OH)COOH$ .***Orthooxybenzoic Acid.*

Salicylic Acid exists naturally in combination in the oils of wintergreen and sweet birch, but is largely prepared from carbolic acid. It is in light, fine, white, prismatic

needles, or in a light, white, crystalline powder; has a very slightly acrid and acid taste, with a not disagreeable sweet after-taste; odorless, but is pungent to the nasal membrane, inducing sneezing; soluble in water, 1:450, alcohol, ether, acetone, glycerin, and chloroform; sparingly soluble in carbon disulfid. A watery solution has an acid reaction to litmus paper, an agreeable taste, and becomes permanently violet colored on addition of 1 drop of ferric-chlorid solution (Reagent 4). The Salicylic Acid in its crystalline state also colors the ferric-chlorid solution. A more reddish color is obtained from neutral salicylates, which color changes to violet, however, on acidulating the fluid with dilute hydrochloric or sulfuric acid.

(1) Salicylic Acid dissolves in cold sulfuric acid (Reagent 21) colorless (if slightly colored, impurities are present); the acid becomes brown colored when boiled with it.

(2) Salicylic Acid dissolves in cold nitric acid (Reagent 15) nearly colorless; with a red color, however, on slightly warming. This solution turns pale yellow when boiled; yellow needles of nitrosalicylic acid form in it by cooling, which are soluble in ammonia-water (Reagent 1) with a red color. The watery solution of nitrosalicylic acid is colored red by an addition of ferric-chlorid solution (Reagent 4); not violet.

(3) The watery saturated solution of Salicylic Acid gives a heavy precipitate with a lead-subacetate solution; not with a neutral 10 per cent lead-acetate solution.

(4) The agreeable odor of wintergreen oil is developed when a previously prepared mixture of 25 c.c. (405 minims) of methyl alcohol with 10 c.c. (162 minims) of sulfuric acid (Reagent 21), in which mixture 0.050 gm. ( $\frac{3}{4}$  gr.) Salicylic Acid is afterwards dissolved, is distilled. The same odor is developed by warming a salicylate with sulfuric acid and wood spirit.

(5) Salicylic Acid in milk may be detected by precipitating fat and proteids with mercuric nitrate and acetic acid, filtering, and agitating the filtrate with ether, which dissolves the Salicylic Acid. After separation the ethereal solution is evaporated and yields the acid in crystals. These are dissolved in alcohol and tested by ferric-chlorid solution (Reagent 4) which gives a violet color; or else they are heated with a mixture of methyl alcohol and sulfuric acid (Reagent 21), when the odor of wintergreen reveals the presence of Salicylic Acid.

**Test for Salipyrin,**  $C_3H(CH_3)_2N_2(C_6H_5)O + C_6H_4 \begin{matrix} \text{OH} \\ \text{COOH} \end{matrix}$ .

*Antipyrin Salicylate.*

Salipyrin is a colorless, crystalline powder, without odor, but possessing the sweetish, acid taste of salicylic acid; soluble (1:25) in water at 100°, 1:200 at 15°; very soluble in chloroform; less so in ether and in alcohol; sparingly soluble in carbon disulfid. The solutions have an acid reaction to litmus paper.

(1) Crystalline tufts are formed and sink to the bottom if 1 c.c. (16 minims) of picric-acid solution (Reagent 16) is added to 10 c.c. (162 minims) of the watery Salipyrin solution.

(2) Salicylic acid is liberated in crystals when 0.20 gm. (3 grs.) Salipyrin is dissolved by warming in 2 c.c. (32 minims) of water and 4 c.c. (64 minims) of hydrochloric acid (Reagent 5) and the fluid allowed to cool.

(3) Salipyrin differs from antipyrin and from salicylic acid in that it gives a dark, fiery-red fluid, changing to violet and blue when warmed with nitric acid (Reagent 15); a black, greasy matter forms thereby in the fluid.

(4) A bluish-green fluid fading to yellow is obtained when 10 drops of acetic acid are mixed with 10 c.c. (162

minims) of a saturated watery solution of Salipyrin, to which 0.10 gm. ( $1\frac{1}{2}$  grs.) sodium nitrite ( $\text{NaNO}_2$ ) has previously been added.

(5) A bluish-green mixture is obtained, which colors red, and in which small crystals of nitrosoantipyrin form, when 0.10 gm. ( $1\frac{1}{2}$  grs.) Salipyrin, 0.10 gm. ( $1\frac{1}{2}$  grs.) sodium nitrite ( $\text{NaNO}_2$ ), and 0.20 gm. (3 grs.) mercuric chlorid ( $\text{HgCl}_2$ ) are agitated together under addition of 4 c.c. (64 minims) of water.

(6) One drop of ferric chlorid (Reagent 4) added to 1 c.c. (16 minims) of a saturated watery solution of Salipyrin, diluted with 9 c.c. (146 minims) of water, gives a reddish-violet-colored fluid.

Test for Salol,  $\text{C}_6\text{H}_4 \begin{smallmatrix} \diagup \text{COO}(\text{C}_6\text{H}_5) \\ \diagdown \text{OH} \end{smallmatrix}$ .

*Phenyl Salicylate.*

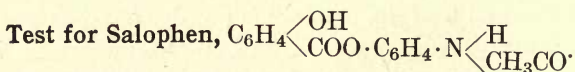
Salol is prepared by heating salicylic acid with phenol, and occurs as a white, crystalline powder of an agreeable, faint, aromatic odor, suggestive of oil of wintergreen, and a sweet taste; it feels gritty between the teeth; is soluble in alcohol, ether, chloroform, benzene, and in fixed or volatile oils; nearly insoluble in water at  $100^\circ$ ; is neutral to litmus paper moistened with alcohol.

(1) 0.005 gm. ( $\frac{1}{200}$  gr.) Salol dissolves with a yellow color in 5 drops of sulfuric acid (Reagent 21) on warming; the color increases in intensity to a pale red when heated nearly to the boiling-point. One drop of ferric-chlorid solution (Reagent 4) colors this solution violet, after it has been diluted with 10 c.c. (162 minims) of water; an agreeable odor develops when this watery dilution is boiled. But if 1 drop of ferric-chlorid solution (Reagent 4) is added to the solution of 0.05 gm. ( $\frac{1}{20}$  gr.) Salol in 1 c.c. (16 minims) of the acid, a dirty green fluid, which soon becomes turbid, is obtained.

(2) Salol dissolves in Reagent 22 with a yellow color reaction.

(3) In its crystalline state Salol does not act upon ferric-chlorid solution (Reagent 4). 0.050 gm. ( $\frac{3}{4}$  gr.) Salol triturated with a glass rod, moistened at the point with a trace of ferric-chlorid solution (Reagent 4), shows no reaction, not even after the addition of 0.5 c.c. (8 minims) of water. The mixture colors violet, however, when 5 c.c. (81 minims) of alcohol is added. 1 drop of ferric-chlorid solution (Reagent 4) increases the color.

(4) A bright greenish-blue solution is obtained when 1 c.c. (16 minims) of sulfuric acid (Reagent 21) is added to a mixture of 0.050 gm. ( $\frac{3}{4}$  gr.) Salol and 0.080 gm. ( $1\frac{1}{4}$  grs.) sodium nitrate ( $\text{NaNO}_3$ ). The mixture is *red* at first, changing to brown, and then to a bluish rather than a green color, when sodium *nitrite* ( $\text{NaNO}_2$ ) is substituted for the nitrate.



*Acetylparaamidophenol. Acetylparaamidophenol Salicylic-ester.*

Salophen is a derivative of salol, and occurs as a white or faint yellowish crystalline powder; odorless and tasteless; having a neutral reaction to litmus paper; sparingly soluble in water; freely soluble in alcohol, ether, chloroform, and in aqueous alkaline solutions; insoluble in carbon disulfid; when heated evolving vapors of a sweetish aromatic odor and of an acid reaction.

(1) A mixture of 0.10 gm. ( $1\frac{1}{2}$  grs.) of Salophen, 0.10 gm. ( $1\frac{1}{2}$  grs.) of chloral hydrate, and 1 c.c. (16 minims) of a 10 per cent caustic-soda solution becomes of a blue or greenish-blue color, gradually turning brown or black; the same reaction will be obtained by using 1 c.c. (16

minims) of chloroform instead of 0.10 gm. ( $1\frac{1}{2}$  grs.) of chloral hydrate; and a somewhat similar color reaction is obtained in a mixture of Salophen, sulfuric acid (Reagent 21), and sodium nitrite ( $\text{NaNO}_2$ ).

(2) If Salophen is boiled with water and some alcoholic potassa solution, a distinct odor of oil of wintergreen is developed on acidifying it with hydrochloric acid (Reagent 5).

(3) Very dilute ferric-chlorid solution added to an alcoholic solution of Salophen develops a violet coloration, rapidly changing to brown-yellow.

(4) If a solution of Salophen in dilute sodium-hydrate solution is boiled and allowed to cool, it soon becomes blue, beginning on the surface. This color is discharged on renewed boiling, and reappears on cooling. When this solution is supersaturated with hydrochloric acid (Reagent 5) and shaken out with ether, a residue is obtained on evaporating the ether, which, dissolved in alcohol, gives a violet color with ferric-chlorid solution (Reagent 4). On adding to the liquid that has been shaken out with ether a little 3 per cent aqueous solution of carbolic acid and some filtered solution of chlorinated lime, the liquid becomes red, and on supersaturation with ammonia-water (Reagent 1) dirty blue.

(5) If 0.10 gm. ( $1\frac{1}{2}$  grs.) Salophen is boiled for one minute with 2 c.c. (32 minims) of solution of sodium hydroxid (1:2), then allowed to cool, and 5 c.c. (81 minims) of solution of chlorinated soda added, a deep and bright-green color is immediately produced; after some time this changes to a deep mahogany-brown. This change to a brown color takes place slowly in the cold, but more rapidly if the solution is boiled. On supersaturating the solution either when green or when brown with a concentrated mineral acid, the color changes to a bright scarlet, and then slowly fades to a red-orange or mandarin color.

**Test for Santonin,  $C_{15}H_{18}O_3$ .**

Santonin is the active principle of wormseed; it is in shining, flattened, prismatic crystals; white when fresh and pale yellow when old; odorless, and nearly tasteless when first put in the mouth, but afterwards developing a bitter taste; nearly insoluble in water; soluble in alcohol, ether, chloroform, acetic acid, olive-oil by warming, and in solutions of caustic alkalies; it has no reaction upon litmus paper. It affects the eyesight when taken internally in large doses (0.20 gm., 3 grs.), causing so-called xanthopsia, when all white objects appear yellow and blue objects green.

(1) 0.005 gm. ( $\frac{1}{12}$  gr.) Santonin agitated with 5 c.c. (81 minims) of a 10 per cent alcoholic solution of caustic potash or soda gives a bright carmine-red-colored fluid, which fades slowly.

(2) Santonin added to sulfuric acid (Reagent 21) and a few drops of ferric-chlorid solution (Reagent 4) and heated gives a red color changing to brown. (This will detect Santonin in the urine.)

(3) 0.010 gm. ( $\frac{1}{6}$  gr.) Santonin evaporated on the water-bath with 1 c.c. (16 minims) of dilute phosphoric acid of 1.154 sp. gr. gives a bright-purplish-red color.

(4) Another characteristic purple-red color reaction of Santonin is obtained when a mixture of 1 gm. ( $15\frac{1}{2}$  grs.) of slaked lime and 1 gm. ( $15\frac{1}{2}$  grs.) of sodium carbonate is moistened with 2 c.c. (32 minims) of alcohol of 94 per cent, and 0.005 gm. ( $\frac{1}{12}$  gr.) Santonin stirred in; the mixture has to be slightly warmed for this reaction; the color is not permanent.

**Test for Sozo-iodol Sodium,  $C_6H_2(OH)I_2 \cdot SO_3Na + 2H_2O$ .***Diiodoparaphenolsulfonic Sodium.*

Sozo-iodol Sodium forms colorless and nearly odorless fine needle-like crystals of a salty taste, suggestive of potassium iodid and with a decidedly sweetish after-taste; is soluble in water, alcohol, glycerin, and chloroform; hardly at all in ether or carbon disulfid; has an acid reaction on litmus paper.

(1) A few drops of bromin-water (Reagent 2), or of chlorin-water (Reagent 3), and 1 drop of ferric-chlorid solution (Reagent 4) added to 5 c.c. (81 minims) of a watery solution (1:100) of Sozo-iodol Sodium, to which previously 1 drop of a freshly prepared gelatinized starch solution has been added, gives immediately the iodine reaction on starch.

(2) A small drop of ferric-chlorid solution (Reagent 4) gives in 50 c.c. ( $1\frac{1}{2}$  f. oz.) of a watery solution (1:100) of Sozo-iodol Sodium a strong violet color reaction.

(3) The watery solution (1:100) soon becomes brown from liberated iodine, if mixed with equal parts of nitric acid (Reagent 15).

**Test for Spartein Sulfate,  $(C_{15}H_{26}N_2)SO_4H_2 + 3H_2O$ .**

Sparteine Sulfate is the salt of a poisonous oily liquid alkaloid obtained from broom, and is described as a white, crystalline powder, odorless, and having a slightly saline and bitter taste; easily soluble in water and in alcohol, and neutral to litmus paper; it dilates the pupil.

(1) A crystal of Sparteine Sulfate, dropped into a drop of solution of ammonium sulfhydrate ( $NH_4HS$ ) causes immediately an orange-red color reaction, that fades somewhat, but reappears permanently when the mixture is spontaneously evaporated to dryness.

(2) Iodin-water (Reagent 6) causes a turbidity in 1 c.c. (16 minims) of Spartein Sulfate solution (45:1000), turning into black crystals.

(3) Spartein is liberated as a colorless oily liquid alkaloid, lighter than water, with an odor faintly suggestive of pyridin, when 10 drops of 10 per cent caustic-soda solution are agitated with 0.050 gm. (8 grs.) of Spartein Sulfate. It is so little volatile at the ordinary temperature, that red litmus paper remains unaffected when a piece is suspended over it in the test-tube in this test; it turns blue, however, when the test-tube is slightly warmed.

#### Test for Strychnin, $C_{21}H_{22}N_2O_2$ .

Strychnin is in colorless, transparent, prismatic crystals, but is usually obtained as a white amorphous powder; odorless, and having an intensely bitter taste perceptible even in highly diluted (1:700000) solution; soluble in water (1:6700), in alcohol (1:110), in chloroform (1:16), in benzene (1:170), in glycerol (1:300), in ether (1:1250); it is also easily soluble in warm turpentine; it has an alkaline reaction upon litmus paper.

(1) On dissolving 0.02 gm. ( $\frac{1}{5}$  gr.) Strychnin in 2 c.c. (32 minims) of nitric acid (Reagent 15), the acid should not turn more than faintly yellow (limit of brucine).

(2) Neither sulfuric (Reagent 21) nor hydrochloric acid (Reagent 5) gives a color reaction in the cold; a yellow or yellow-brown color is only to be noticed on warming.

(3) A bright blue-violet color reaction, changing to red, is obtained when Strychnin is dissolved in sulfuric acid (Reagent 21), or in concentrated phosphoric acid (sp. gr. 1.7), and oxidizing agents are added, such as potassium permanganate, chromic or hydriodic acid, potassium dichromate, potassium ferricyanid, peroxid of lead and of manganese, or cerous hydroxid.

To succeed with this reaction dissolve a small particle of Strychnin, or of one of its salts, in 1 or 2 drops of sulfuric acid (Reagent 21) or of concentrated phosphoric acid on a porcelain lid; throw a few small particles of any of the above-named oxidizing agents upon the acid, and move the lid slowly to and fro; or throw a particle of Strychnin upon a few drops of sulfuric acid containing chromic acid (Reagent 22).

All the oxidizing agents named are, of course, not equally sensitive, and the most successful color reactions are obtained with dilute solutions or with fractions of a milligram. Potassium permanganate is the most sensitive reagent. The color reaction of potassium permanganate with sulfuric acid alone cannot be mistaken for a Strychnin reaction; the blue-violet streaks of a Strychnin color reaction being very characteristic. It is preferable to employ sulfuric acid containing potassium permanganate (Reagent 23), and not potassium permanganate in substance. Cerous hydroxid has a great advantage over potassium permanganate and over potassium dichromate in that it is white. Dissolve cerium oxalate in 10 per cent dilute sulfuric acid, precipitating with ammonia of 30 per cent, filtering and washing. The moist cerous hydroxid gives a beautiful color reaction in 1:100,000 of Strychnin, changing to rose-pink.

No Strychnin reaction appears with sulfuric acid containing chromic acid (Reagent 22) when brucin and Strychnin, mixed in equal parts, are submitted to the test; it succeeds, however, in this proportion with sulfuric acid containing potassium permanganate (Reagent 23). Morphin obscures the reaction as follows: A solution of 0.00001 gm. ( $\frac{1}{64000}$  gr.) Strychnin, evaporated with a solution of 0.001 gm. ( $\frac{1}{64}$  gr.) morphin sulfate on a water-bath, yields a blurred, Strychnin reaction,

when the residue is dissolved in sulfuric acid (Reagent 21) and a crystal potassium permanganate added.

(4) Evaporate 1 c.c. (16 minims) of fluid extract nux vomica to dryness; add 2 c.c. (32 minims) water to the residue; stir with a glass rod; filter; dilute filtrate with an equal proportion of water; divide a few drops over a large surface on porcelain lids; evaporate to dryness, and add to one residue with a glass rod 1 drop of sulfuric acid containing chromic acid (Reagent 22), to another 1 drop sulfuric acid containing potassium permanganate (Reagent 23). A blue-violet reaction, immediately disappearing, takes place.

### Test for Sugar in Urine.

(1) Acidulate the urine with acetic acid, boil, and filter out any albumin if necessary. Then mix the filtrate with equal parts of liquor potassæ and heat to boiling, when ordinary urine will turn brownish red, but saccharine urine will become dark brown or black. (Moore's test.)

(2) Dissolve 2.5 gm. ( $38\frac{1}{2}$  grs.) of *pure* bismuth oxynitrate (free especially from silver) and 4 gms. ( $61\frac{1}{2}$  grs.) of Rochelle salt in 100 gms. (1543 grs.) of 8 per cent solution of sodium hydrate, and preserve for use. To use this reagent 1 c.c. (16 minims) of urine is added to 10 c.c. (162 minims), and the whole boiled gently for some time, when, if even only traces of sugar be present, the mixture becomes black. (Boettger's test.)

(3) Render the urine alkaline with potassium hydrate and filter to remove any phosphates, etc., which may precipitate. To the filtrate add 5 or 10 drops of solution of sulfate of copper; pour in solution of potash or soda until the precipitate first formed is re-dissolved; slowly heat the solution to near the boiling-point; a

yellow, yellowish-red, or red precipitate (cuprous oxide) is formed if sugar be present.

(4) Place in a test-tube 30 minims of water with an indigo and a sodium-carbonate paper. Heat the test-tube gently until the indigo is dissolved. (The solution should be only a pale-blue color. A portion of one of the indigo papers may suffice, but the whole of the soda paper should be used.) Add to the blue solution from a pipette 1 drop of the urine to be tested and keep the fluid at a boiling-point, without, however, permitting active ebullition, for sixty seconds. If no change is produced, add a second drop of the urine, and heat once more. If any notable quantity of sugar is present the fluid will be observed to change from pure blue to violet, then to purple and red; with only a trace of sugar the color will merely change to one of the intermediate shades. (Mulder's test.)

(5) Mix in a test-tube equal volumes of No. 1 and No. 2 Fehling's solution, dilute with an equal quantity of water, and boil. No precipitate should be noticed. Now add to this mixture half its volume of urine and again boil. The presence of glucose will be indicated by an orange or red-colored precipitate. Fehling's solution is made as follows: In one bottle keep the following, No. 1: pure copper sulfate 17.32 gms. (267 grs.); distilled water 250 c.c. ( $8\frac{1}{2}$  f. oz.). In another bottle, No. 2: Rochelle salt 87.00 gms. (3 oz. and 30 grs.); caustic soda 25.00 gms. (386 grs.); distilled water 250 c.c. ( $8\frac{1}{2}$  f. oz.)

#### Test for Sulfonal, $(\text{CH}_3)_2\text{C}(\text{SO}_2 \cdot \text{C}_2\text{H}_5)_2$ .

*Dimethylmethandiethylsulfon. Disulfonethyldimethylmethan.  
Diethylsulfondimethylmethan.*

Sulfonal is a colorless crystalline powder; inodorous and nearly tasteless; soluble in water (1:500), in alcohol, in

ether, in chloroform, and in carbon disulfid; neutral to litmus paper.

(1) An odor suggestive of garlic (mercaptan) and likewise acid vapors are liberated when 0.050 gm. ( $\frac{3}{4}$  gr.) Sulfonal is heated, very nearly to charring, in a glass tube with 1 gm. ( $15\frac{1}{2}$  grs.) of water-free sodium acetate, proving the presence of sulfur. A farther reaction on sulfur in Sulfonal is to cover the test-tube in which the latter has been melted with dry sodium acetate, with a piece of filter-paper, previously moistened with a solution of 0.10 gm. ( $\frac{1}{6}$  gr.) nitroprussid sodium in 10 c.c. (162 minims) of 10 per cent ammonia; this paper becomes red, violet, and blue, the colors soon fading. Or substitute a piece of filter-paper moistened with a 10 per cent lead-acetate solution, for the nitroprussid sodium paper, which will become black. Or melt 0.10 gm. ( $\frac{1}{6}$  gr.) Sulfonal with 0.20 gm. ( $\frac{1}{3}$  gr.) of potassium cyanid in a test-tube, notice the odor suggestive of garlic (mercaptan odor); cool; dissolve the slack in a few centimeters of water; acidulate with hydrochloric acid (Reagent 5) and add a drop of ferric-chlorid solution (Reagent 4); this gives a blood-red color reaction (sulfocyanate of iron).

(2) Mixed with powdered charcoal and heated in a test-tube, Sulfonal is reduced and breaks up into mercaptan (detected by garlicky odor), formic and acetic acids (the vapor reddens litmus paper), and sulfur dioxid (which bleaches paper wet with blue-starch iodid).

### **Test for Terpin Hydrate, $C_{10}H_{18}(OH)_2 + H_2O$ .**

Terpin Hydrate occurs in large colorless crystals, with a very slight aromatic odor suggestive of pine leaves, and a faintly bitter taste; is easily soluble in warm glacial acetic acid; in alcohol of 0.825 sp. gr.; sparingly soluble in ether, chloroform, and carbon disulfid; in water

1:250. Its watery solution does not act upon litmus paper.

(1) Terpin Hydrate gives a red-yellow color reaction when strewn upon sulfuric acid (Reagent 21), thereby developing an agreeable aromatic odor.

(2) Nitric acid (Reagent 15) does not act upon Terpin Hydrate in the cold, but a violent action takes place by warming Terpin Hydrate with it.

(3) A rose-red color reaction is given when Terpin Hydrate is evaporated in an open dish on the water-bath with a 10 per cent sulfuric acid.

(4) Terpin Hydrate gives a yellow color reaction, changing to brown and black-brown when strewn upon a freshly made mixture of 0.020 gm. ( $\frac{1}{5}$  gr.) ammonium molybdate and 5 drops sulfuric acid (Reagent 21), spread out in a thin layer. A red to brown color reaction takes place if bismuth subnitrate is substituted for the ammonium molybdate. Titanic acid gives yellow to gold-colored tinges if taken instead, and ammonium tungstate gives yellow, brown, gray, or lilac colors.

(5) A yellow color is obtained when a mixture of 0.005 gm. ( $\frac{1}{20}$  gr.) Terpin Hydrate and 0.10 gm. ( $1\frac{1}{2}$  grs.) sugar are triturated with 3 drops of sulfuric acid (Reagent 21); this color fades by and by to a rose hue.

**Test for Thallin Sulfate,  $(C_{10}H_{13}NO)_2H_2SO_4 + 2H_2O$ .**

*Sulfate of Oxiquinolintetrahydromethylether. Sulfate of Tetrahydroparaquin methylphenate.*

Thallin Sulfate is the salt of an artificial alkaloid of the quinolin series, and is a somewhat yellowish or brownish, not strictly white, crystalline powder with an agreeable odor suggestive of Tonka bean; is soluble in 1:7 parts of water, with a bitter, salty taste and an acid

reaction to litmus paper; also soluble in alcohol and in warm chloroform.

(1) Thallin Sulfate colors chlorin-water (Reagent 3) green if strewn upon it; green flocks soon sink to the bottom; the mixture turns violet. The same green coloring takes place with bromin-water (Reagent 2); the precipitate is brown colored, however, redissolving with a green color on addition of more of the Thallin Sulfate.

(2) 1 c.c. (16 minims) of ferric-chlorid solution (Reagent 4) gives a green color, in the course of an hour, in 0.5 c.c. (8 minims) of a 1 per cent solution of Thallin sulfate diluted with 500 c.c. (17 f. oz.) of water. The color appears sooner and more brightly when more Thallin Sulfate and less water is taken. The green fluid fades to a red and a yellow-brown color. A 1 per cent watery solution of Thallin Sulfate becomes by itself, without any addition, greenish-brown colored when exposed to light and air.

(3) A brown filtrate is obtained which colors the filter-paper reddish when 0.005 gm. ( $\frac{1}{12}$  gr.) of Thallin Sulfate is agitated with 0.005 gm. ( $\frac{1}{12}$  gr.) of a good commercial peroxid of manganese and 3 c.c. (49 minims) of water. The solution turns to a bright green when a few drops of acetic acid are added, which color changes to brown and red; the red fluid is fluorescent.

(4) Thallin Sulfate dissolves colorless in sulfuric acid (Reagent 21), but a permanent red color reaction sets in if a trace of nitric acid (Reagent 15) is mixed with the sulfuric acid.

(5) Thallin salts cause dark coloration of urine when given by the stomach or subcutaneously.

(6) A green fluid changing to red is obtained if 0.005 gm. ( $\frac{1}{12}$  gr.) of Thallin Sulfate are dissolved in 2 c.c. (32 minims) of water with 0.050 gm. ( $\frac{1}{2}$  gr.) of potassium iodate ( $\text{KJO}_3$ ) or of potassium ferricyanid,  $\text{K}_3\text{Fe}(\text{CN})_6$ .

**Test for Thebain,  $C_{17}H_{15}NO(OCH_3)_2$ .**

Thebain is a poisonous alkaloid found in opium; it is a white, crystalline powder of an acrid and styptic rather than bitter taste; is scarcely soluble in water; easily soluble in alcohol, chloroform, and carbon disulfid; less soluble in ether; has a faint alkaline reaction when laid upon red litmus paper moistened with water; this reaction is sharper and appears sooner if the alkaloid is moistened with alcohol.

(1) Thebain colors chlorin-water (Reagent 3) red when it is moistened with a small quantity of the reagent; this red color fades to yellow when more of the reagent is used.

(2) On nitric acid (Reagent 15) Thebain gives a yellow color reaction.

(3) On sulfuric acid (Reagent 21) Thebain gives a permanent bright-red color reaction.

(4) On hydrochloric acid (Reagent 5) Thebain gives a greenish-yellow color reaction.

(5) The blue color which morphin gives with ammonium molybdate and sulfuric acid is brown when Thebain is treated as described for morphin and when bis-muth subnitrate, titanio acid, or tungstic acid is used, changing to green with the molybdate.

**Test for Theobromin,  $C_5H_2(CH_3)$ .***Dimethylxanthin.*

Theobromin occurs in the seed of *Theobroma cacao*, from which chocolate is made; it is a light, white, somewhat crystalline powder of a bitter taste; is soluble in 1:70 parts of water at 100°; very little soluble in alcohol, in ether and in benzene; slightly in chloroform and in car-

bon disulfid; fairly soluble in glacial acetic acid; it does not act upon litmus paper.

(1) 0.020 gm. ( $\frac{1}{2}$  gr.) Theobromin dissolves in 2 c.c. (32 minims) of ammonia-water (Reagent 1) by slightly warming; small, white, crystalline needles form in this solution in the course of a few hours if 2 drops of a saturated watery solution of silver nitrate are added, and the mixture kept in a loosely corked test-tube in a water-bath.

### Test for Thymol, $C_6H_3 \cdot CH_3(OH)C_3H_7$ .

#### *Propylmetakresol.*

Thymol is a phenol occurring in the volatile oil of thyme, and is described as in large, colorless, translucent crystals of the hexagonal system, having a strong, aromatic odor of thyme and a pungent, hot taste, with a very slight caustic effect upon the lips; they develop electricity when rubbed, and attract small pieces of paper; very slightly soluble in water 1:1000; easily soluble in alcohol, ether, chloroform, acetic acid, carbon disulfid, warm glycerol, and in fixed or volatile oils; and liquefy when triturated with camphor, menthol, or chloral hydrate; they sink in water.

(1) An intense pink-colored fluid of a permanent color is obtained when 1 c.c. (16 minims) of chloroform U. S. P.—containing 1 per cent of alcohol—is poured upon a mixture of 0.010 gm. ( $\frac{1}{2}$  gr.) Thymol and 0.010 gm. ( $\frac{1}{2}$  gr.) caustic potash or soda.

(2) A turbid, violet-colored mixture is obtained when a mixture of 0.005 gm. ( $\frac{1}{2}$  gr.) Thymol and 0.010 gm. ( $\frac{1}{2}$  gr.) potassium nitrite or sodium nitrite is warmed in a test-tube for about ten minutes to  $75^\circ$  or  $80^\circ$  with 1 c.c. (16 minims) of mercuric-chlorid solution (Reagent 13) diluted with 2 c.c. (32 minims) of water.

(3) An intense, permanent bluish-green color reaction takes place when a small particle of Thymol is dropped into a mixture of 1 c.c. (16 minims) of glacial acetic acid, 4 drops of sulfuric acid (Reagent 21), and 1 drop of nitric acid (Reagent 15); camphor and menthol give no color reaction when similarly treated.

(4) 0.50 gm. (8 grs.) Thymol, triturated with twelve drops sulfuric acid (Reagent 21), give a yellowish-red fluid, solidifying in about four hours to a hard carmine-red, crystalline mass. A small particle of this red compound dissolved in 50 c.c. ( $1\frac{1}{2}$  f. oz.) of water to which previously 1 drop of ferric-chlorid solution (Reagent 4) has been added colors the water violet.

### Test for Tritopin, $C_{42}H_{54}N_2O_7$ .

Tritopin is an alkaloid found in opium; it crystallizes in characteristic anhydrous, transparent, needle-like plates; easily soluble in chloroform; less so in alcohol of 94 per cent and in 10 per cent caustic-soda solution; sparingly soluble in ether; insoluble in carbon disulfid; gives a blue spot on moist, sensitive, red litmus paper.

(1) Tritopin gives a rose-red color reaction when strewn upon sulfuric acid (Reagent 21), which color increases in time. The color changes to green, blue, and violet by warming on the water-bath or over a flame.

(2) A bright violet-red color reaction is obtained if a trace of ferric-chlorid solution (Reagent 4) is added to the solution of Tritopin in sulfuric acid (Reagent 21) with a glass rod. The color is dark red when a trace of sodium nitrate ( $NaNO_3$ ) is substituted for the ferric-chlorid solution (Reagent 4).

(3) Tritopin gives a brown color reaction when treated with sulfuric acid and bismuth subnitrate, as described for morphin, which brown color changes to red and then

to yellow-brown. With ammonium molybdate and sulfuric acid and with titanous acid it gives a lilac color.

### Test for Urethan, $\text{NH}_2 \cdot \text{CO} \cdot \text{O} \cdot \text{C}_2\text{H}_5$ .

#### *Ethylcarbamate.*

Urethan is made by allowing ammonia to act upon ethylcarbamate; it forms large, colorless crystals of a faint ethereal odor and taste suggestive of saltpetre which are soluble in water, ether, chloroform, alcohol, and in carbon disulfid by warming; has no action upon litmus paper.

(1) It liberates ammonia ( $\text{NH}_3$ ) when 0.100 gm. ( $1\frac{1}{2}$  grs.) Urethan is dissolved in 10 drops of sodium-hydrate solution (Reagent 20) or in 10 c.c. (162 minims) of lime-water (Reagent 10), slowly in the cold, more quickly by warming.

(2) Iodoform is formed when 0.10 gm. ( $1\frac{1}{2}$  grs.) of iodine, in small portions, is added to a solution of 0.20 gm. (3 grs.) of Urethan dissolved in 2 c.c. (32 minims) of a warm 10 per cent caustic-soda solution. If the well-known, fine yellow crystalline precipitate of iodoform does not form at once, a few particles more of iodine will have to be added to the solution.

### Test for Vanillin, $\text{C}_6\text{H}_3\text{CHO}(\text{OCH}_3)\text{OH}$ .

#### *Methylprotocatechnaldehyd.*

Vanillin is the odoriferous principle of the vanilla bean, which contains from 2 to  $2\frac{1}{2}$  per cent. It is also formed synthetically when guaiacol is acted upon with chloroform and alkali; is in crystalline needles of a powerful aromatic odor and taste; soluble in water 1:100; easily soluble in alcohol, ether, chloroform, and carbon disulfid.

(1) A small particle of Vanillin added to a solution of 0.010 gm. ( $\frac{1}{8}$  gr.) of pyrogallol in 1 c.c. (16 minims) of hydrochloric acid (Reagent 5) heated to boiling gives a bright violet-red color reaction.

(2) A blue fluid is obtained when 0.020 gm. ( $\frac{1}{5}$  gr.) of Vanillin is agitated with 2 c.c. (32 minims) of water to which 1 drop ferric-chlorid solution (Reagent 4) has been added. The blue color turns brown when the fluid is warmed.

(3) Vanillin dissolves in sulfuric acid (Reagent 21) with a yellow color; it colors nitric acid (Reagent 15) yellow-red when strewn upon it.

### Test for Veratrin.

Veratrin is a mixture of alkaloids obtained from cevadilla. It is a white or grayish white, amorphous, or semi-crystalline powder, odorless, but causing intense irritation and sneezing when even a minute quantity reaches the nasal mucous membrane; has an acrid taste, and leaves a sensation of tingling and numbness on the tongue; is easily soluble in alcohol, chloroform, and ether; soluble in benzene and carbon disulfid; very slightly in water 1:1500; on moist red litmus paper it gives a blue spot.

(1) 0.005 gm. ( $\frac{1}{20}$  gr.) of Veratrin added to 5 c.c. (81 minims) sulfuric acid (Reagent 21) becomes lumpy and yellow colored; violet on warming the solution; this color changes to orange and cherry-red, and the acid slowly becomes thus colored.

(2) 0.005 gm. ( $\frac{1}{20}$  gr.) Veratrin gives no color reaction in the cold with 5 c.c. (81 minims) hydrochloric acid (Reagent 5); by warming it over the flame the solution colors pink-red; 0.10 gm. ( $1\frac{1}{2}$  grs.) Veratrin boiled with 5 c.c. (81 minims) of the acid gives a purple-red color reaction.



(3) 0.10 ( $1\frac{1}{2}$  grs.) Veratrin warmed on the water-bath with 1 c.c. (16 minims) of phosphoric acid of 1.4 sp. gr. colors purple-red, and gives off an odor suggestive of butyric acid.

(4) A dark-green color reaction, followed by reddish purple and blue colors, is obtained by adding a sprinkling of finely powdered sugar to a solution of Veratrin in sulfuric acid (Reagent 21).

### Test for Yohimbine, $C_{23}H_{49}N_2O_4$ .

Yohimbine is an alkaloid obtained from the bark of *Coryanthe yohimbi*, a large tree growing in the southwestern part of Africa. It is in the form of white needles, but turns yellow, then orange-red, when exposed to the air; has a bitter taste, and is soluble in water and alcohol; melts at  $232^{\circ}$  C.

(1) When Yohimbine is treated with a solution of gold chloride (1:100) an amorphous, grayish-violet precipitate is formed.

(2) Sulfuric acid (Reagent 21) added to a solution of Yohimbine develops the odor of mint.

(3) Nitric acid (Reagent 15) followed by caustic potassa produces a green color with the acid, changed to cherry-red by the potassa when in contact with Yohimbine.

(4) Reduction of silver takes place when a solution of Yohimbine is mixed with a solution of silver nitrate.

(5) By oxidation with potassium permanganate Yohimbine yields yohimbic acid ( $C_{20}H_{24}N_2O_6$ ) and noryohimbic acid ( $C_{19}H_{20}N_2O_7$ ).

(6) Yohimbine gives with cane-sugar, glucose, or furfural on addition of sulfuric acid (Reagent 21) the same color reactions, red or violet, that are given by the biliary acids, but this color reaction cannot be regarded as ser-

viceable for its identification, as sesame oil produces the same reaction.

### Test for Zinc Sulfate, $\text{ZnSO}_4 + 7\text{H}_2\text{O}$ .

Zinc Sulfate is in colorless, transparent, rhombic crystals, or in a granular crystalline powder, without odor, and having an astringent, metallic taste. Efflorescent in dry air. Soluble in 0.6 part of water at  $15^\circ \text{C}$ ., and in 0.2 part of boiling water, also soluble in 3 parts of glycerin, but insoluble in alcohol. Its aqueous solution shows an acid reaction to blue litmus paper.

(1) The aqueous solution of Zinc Sulfate (1 in 20) yields a white gelatinous precipitate with potassium ferrocyanid T.S., and a white precipitate with ammonium sulfid T.S., and with barium chlorid T.S.

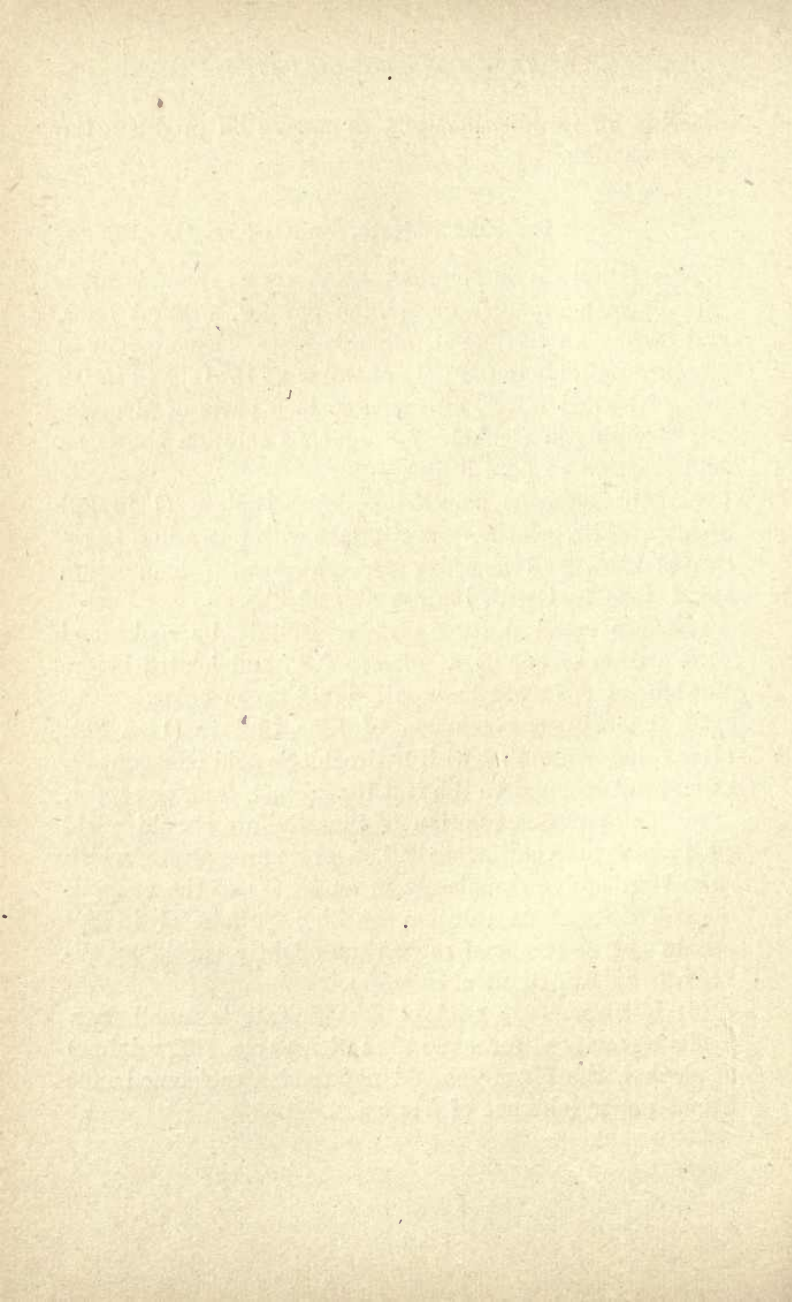
(2) If a small portion of Zinc Sulfate be moistened with a drop of cobaltous nitrate T.S., and heated before the blowpipe, it will assume a vivid green color.

(3) The aqueous solution of Zinc Sulfate (1 in 20), after being acidulated with hydrochloric acid (Reagent 5), should not respond to the test for *arsenic*, *lead*, or *copper*.

(4) The aqueous solution of Zinc Sulfate should yield with ammonium carbonate T.S., a white precipitate, which should redissolve completely in an excess of the reagent.

(5) The aqueous solution of Zinc Sulfate (1 in 20) should not be rendered more than slightly turbid by silver nitrate T.S. (limit of *chloride*).

(6) If 1 gm. ( $15\frac{1}{2}$  grs.) of Zinc Sulfate in small fragments be agitated for some time with 10 c.c. (162 minims) of alcohol, the filtrate should not redden moistened blue litmus paper (absence of *free acid*).



## TESTS FOR PURITY.

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### Tests for the Presence and Purity of Alcohol, $C_2H_5OH$ .

Alcohol is a transparent, colorless, mobile, and volatile liquid of a slight, agreeable odor and a burning taste. Specific gravity about 0.816 at  $15.6^{\circ}C$ . Miscible with water in all proportions, and without any trace of cloudiness; also miscible with ether or chloroform. It is readily volatilized, even at low temperatures, and boils at  $78^{\circ}C$ . It is inflammable, and burns with a pale-blue, smokeless flame. It should not affect the color of blue or red litmus paper previously moistened with water; and if evaporated in a clean glass vessel, no color or weighable residue should remain.

(1) If 10 c.c. (162 minims) of Alcohol be mixed with 5 c.c. (81 minims) of water and 1 c.c. (16 minims) of glycerin, and the mixture allowed to evaporate spontaneously from a piece of clean, odorless blotting-paper, *no foreign odor* should become perceptible when the last traces of the Alcohol leave the paper (absence of *fusel-oil constituents*).

(2) If 25 c.c. (405 minims) of Alcohol be allowed to evaporate spontaneously in a porcelain evaporating-dish, carefully protected from dust, until the surface of the dish is barely moist, no red or brown color should be produced upon the addition of a few drops of colorless, concentrated sulfuric acid (Reagent 21) (absence of *amyl*

*alcohol*, or *non-volatile*, *carbonizable*, *organic impurities*, etc.).

(3) If 10 c.c. (162 minims) of Alcohol be mixed in a test-tube with 5 c.c. (81 minims) of potassium hydroxid T.S., the liquid should not at once assume a yellow color (absence of *aldehyde* or *oak tannin*).

(4) If 20 c.c. (324 minims) of Alcohol be shaken in a clean, glass-stoppered vial with 1 c.c. (16 minims) of silver nitrate T.S., the mixture should not become more than faintly opalescent, or acquire more than a faint brownish tint when exposed for six hours to diffused daylight (limit of *organic impurities*, *amyl alcohol*, *aldehyde*, etc.).

(5) Into a test-tube of the capacity of about 40 c.c. (1 f. oz., 169 minims) 1 c.c. (16 minims) of the Alcohol or spirit to be tested should be poured, and, if it be undiluted, enough distilled water added to make the liquid measure 10 c.c. (162 minims). If the Alcohol be already diluted, a correspondingly larger volume of it should be taken and diluted to 10 c.c. (162 minims), so that the proportion of Alcohol in the liquid shall not be more than about 10 per cent by volume. A copper-wire spiral (made by winding 1 meter (39 inches) of No. 18 clean copper wire closely around a glass rod 7 millimeters ( $\frac{28}{100}$  inch) thick, making a coil about 3 centimeters (1 inch) long, the end of the wire being formed into a handle) should be heated to redness in a flame free from soot and plunged quite steadily to the bottom of the liquid in the test-tube and held there for a second or two, then withdrawn and dipped into water to cool. This treatment with red-hot copper should be repeated five or six times, immersing the test-tube in cold water to keep down the temperature of the liquid. The contents of the test-tube should now be filtered into a wide test-tube and boiled very gently. If the odor of acetalde-

hyde be perceptible, the boiling is to be continued until the odor ceases to be distinguished clearly. The liquid is now cooled, and to it should be added 1 drop of a solution containing 1 part of resorcinol in 2000 parts of water. A portion of this liquid is then poured cautiously into a second test-tube containing pure sulfuric acid (Reagent 21) in such a way that the two liquids shall not mix, the tube being held in an inclined position; this tube is allowed to stand for three minutes and then slowly rotated. No rose-red ring should show at the line of contact of the two layers (absence of more than 2 per cent of *methyl alcohol*).

(6) Filter the liquid to be tested, if not clear, and warm it in a test-tube; add a little iodine and a few drops of potassium or sodium-hydrate solution; again warm gently and set aside for a time; a yellowish crystalline deposit of iodoform ( $\text{CHI}_3$ ) is obtained if Alcohol is present. This is Lieben's test, and will detect 1 part of Alcohol in 2000 parts of water.

(7) Add 0.10 gm. ( $1\frac{1}{2}$  grs.) of potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) and 1 c.c. (16 minims) of sulfuric acid (Reagent 21) to the suspected liquid and heat it; a green color and the odor of aldehyde reveal the presence of Alcohol.

(8) Heat the liquid to be tested in a test-tube with a few grains of sodium acetate and 3 or 4 drops of sulfuric acid (Reagent 21); the odor of apples (acetic ether) is developed if it contains Alcohol.

(9) A drop of benzoyl chlorid ( $\text{C}_6\text{H}_5\text{COCl}$ ) shaken with an alcoholic liquid and warmed with sodium-hydrate solution (Reagent 20) gives off the odor of ethyl benzoate (benzoic ethyl).

(10) Alcohol in the urine is tested by first distilling the urine, then dissolve, by heating, 1 part, by weight, of molybdic acid in 2 parts sulfuric acid (Reagent 21);

add a drop of this to the distillate; a blue coloration indicates Alcohol.

(11) To detect Alcohol in essential oils strongly agitate the oil with an equal volume of distilled water; remove the watery layer when clear, and add a drop or two of the solution of molybdic acid in sulfuric acid (10); an azure-blue coloration is developed if it contains a trace of Alcohol.

(12) Add solution of mercuric nitrate U.S.P. to a liquid containing Alcohol; a partial reduction to mercurous nitrate takes place; after the action is complete add ammonia-water (Reagent 1); a black precipitate is formed (difference from *methyl alcohol*).

(13) The presence of Alcohol in chloroform and in chloral hydrate is ascertained by adding, very carefully and gradually, to the chloroform or to an aqueous solution of the chloral hydrate a solution of molybdic acid in sulfuric acid (10), when the presence of the smallest trace of Alcohol will cause a blue coloration.

(14) The fluid to be tested is carefully poured upon concentrated nitric acid (Reagent 15) contained in a test-tube. In the presence of Alcohol a greenish coloration soon becomes apparent at the point of contact of the two fluids, which soon becomes intensely emerald-green. Gradually, also, faint evolution of gas occurs, which is accompanied by the odor of amyl nitrite.

(15) Heat 10 c.c. (162 minims) of an alcoholic liquid with about one-fourth its volume of sulfuric acid (Reagent 21); the odor of ether will be evolved.

(16) Mix the liquid to be tested with platinum black, and put it in a small flask; heat the flask to  $51.1^{\circ}\text{C.}$ ; shake it well, and filter the liquid; to the filtrate add a few drops of potassium-hydrate solution, and evaporate to dryness in a water-bath; heat the residue with a little arsenic trioxid ( $\text{As}_2\text{O}_3$ ); when, if Alcohol is present, a

garlicky odor will be noticed, owing to the production of cacodyl ( $\text{As}_2(\text{CH}_3)_4$ ).

(17) Alcohol in chloroform may be detected by shaking the chloroform with potassium permanganate and potassium-hydrate solution, made by dissolving 1 part of potassium permanganate and 10 parts of potassium hydrate in 250 parts of water; which gives a violet color changing to green.

(18) A delicate test for Alcohol in very dilute solution, by which 1 part may be recognized in 1,000,000 parts of water, depends on the formation of aldehyde by distilling the weak solution of Alcohol with sulfuric acid containing chromic acid (Reagent 22). If to the distillate a few drops of fuchsin solution, which has been decolorized by sulfur dioxid, are added, a violet coloration is produced, the intensity of which depends on the amount of aldehyde present. The color is then compared with a solution of potassium permanganate, the color of which is adjusted to that produced by the fuchsin reagent in an aldehyde solution of known strength.

### Test for the Purity of Allspice.

(1) Wash the allspice thoroughly and evaporate the wash-water slowly to one-fourth its volume; add a few drops of nitric acid (Reagent 15) and stir with a glass rod, then add, drop by drop, a solution of potassium ferrocyanid; a blue color indicates ferric oxide or Armenian bole as the adulterant.

(2) A half teaspoonful of the sample is stirred into half a cupful of boiling water, and the boiling continued for two or three minutes. The mixture is then cooled. Dilute with an equal volume of water. A single drop of tincture of iodine is now added. If starch is present,

a deep-blue color, which in the presence of a large amount of starch appears black, is formed.

### Test for the Purity of Almond-oil.

(1) If 2 c.c. (32 minims) of Almond-oil be vigorously shaken with 1 c.c. (16 minims) of fuming nitric acid and 1 c.c. (16 minims) of water, a whitish (not red or brownish) mixture should be formed, which, after standing for some hours at about 10° C., should separate into a solid, white mass, and a scarcely colored liquid (distinction from fixed oils of *apricot* and *peach kernels*, and from *sesame*, *cottonseed*, and *poppy-seed oils*).

(2) If 10 c.c. (162 minims) of the oil be mixed with 15 c.c. (4 fluid drachms) of a 15 per cent solution of sodium hydrate and 10 c.c. (162 minims) of alcohol, and the mixture allowed to stand at a temperature of 35° to 40° C., with occasional agitation, until it becomes clear, and then diluted with 100 c.c. (3 f. oz.) of water, the clear solution thus obtained, upon the subsequent addition of an excess of hydrochloric acid (Reagent 5), will set free a layer of oleic acid. This, when separated from the aqueous liquid, washed with warm water, and clarified on a water-bath, will remain liquid at 15° C., although sometimes depositing particles of solid matter and becoming turbid. One part of this oleic acid, when mixed with 1 volume of alcohol of 0.820 specific gravity, should give a clear solution, which at 15° C. should not deposit any fatty acids, nor become turbid on the further addition of 1 volume of alcohol (distinction from *olive*, *arachis*, *cottonseed*, *sesame*, and *other fixed oils*).

### Test for the Purity of Ambergris.

(1) Pure Ambergris has a specific gravity of 0.908 to 0.920, and is insoluble in water; very slightly soluble

in cold, but more readily soluble in hot alcohol, in ether, and in volatile and fatty oils. In absolute alcohol it is almost completely soluble. It contains 85 per cent of ambrein, and on distillation with water yields 13 per cent of a volatile, pleasantly odorous oil. On account of its high price Ambergris should be tested for the presence of benzoin, olibanum, wax, flour, and similar substances.

### Test for the Purity of Animal Charcoal.

(1) Place a weighed quantity of the suspected charcoal, previously finely powdered and dried, in a porcelain crucible, and heat until all organic substances have been incinerated. Not more than *one-tenth* of the weight should be lost by this operation.

(2) Boil the sample several times with pure water, dry, add a small quantity of potassium hydrate, and bring again to the boiling-point. After a few minutes filter. If the filtrate appears colored, the charcoal has already been used, and not thoroughly revived.

### Test for the Purity of Arrowroot.

(1) The presence of potato-starch in Arrowroot may be discovered by the microscope. Arrowroot consists of regular ovoid particles of nearly equal size, whereas potato-starch consists of particles of an irregular ovoid or truncated form, exceedingly irregular in their dimensions, some being so large as  $\frac{1}{300}$  of an inch, and others only  $\frac{1}{2000}$ . Arrowroot may be distinguished from potato-starch not only by the different size of its particles, but by the difference of structure. Their surfaces in the Arrowroot are smooth, and free from the streaks and furrows seen in the potato particles by a good microscope. The Arrowroot, moreover, is destitute of that

fetid unwholesome oil extractable by alcohol from potato-starch.

(2) Triturate the sample with dilute nitric acid 1.10 in a mortar. If it immediately forms a transparent very viscid paste or jelly, it is adulterated with potato-starch or flour. Pure Arrowroot forms an opaque paste, and takes a much longer time to become viscid.

### **Test for the Purity of Asphaltum.**

(1) Dissolve the sample in bisulphid of carbon, filter, evaporate to dryness, and heat until it can be rubbed to a fine powder in a mortar. One part by weight of this is gently digested with 50 parts of sulphuric acid (Reagent 21) for twenty-four hours, and then gradually with 100 parts of water, and allowed to cool thoroughly. This mixture is filtered and diluted with 1000 parts of water. The unadulterated Asphaltum gives a colorless or pale-yellow fluid, while, if pitch, coal-tar, etc., are present, it is dark brown or black.

### **Test for the Purity of Beeswax.**

(1) Pure Beeswax should have a specific gravity of 0.959 to 0.967 and a melting-point of 63° to 64° C.

(2) Its composition is approximately constant, and the average amount of potassium hydrate required is: for neutralization of the free acids, 2 per cent; for saponification of esters, 7.5 per cent; or a total of 9.5 per cent of potassium hydrate for complete saponification.

(3) The presence of stearic acid is confirmed by boiling the wax in alcohol, filtering when cold, and pouring it into water, when a flocculent precipitate is immediately formed.

(4) Boil the wax with an aqueous solution of sodium

carbonate, an effervescence indicates the presence of stearic acid as the adulterant.

(5) Pure wax floats in alcohol of 29°. By observing the strength of the alcohol in which the sample floats, the percentage of wax may be deduced as follows:

If the alcoholometer shows 29°, the sample contains 100 per cent of wax; 39.63°, 75 per cent; 50.25°, 50 per cent; 60.87°, 25 per cent; 71.50°, 0 per cent, no wax at all, but tallow.

### Test for the Purity of Benzine, $C_5H_{12}C_6H_{14}$ .

(1) Pure Benzine on calendered white writing paper will evaporate, free of stain, in seven minutes.

(2) Commences to boil at 54° to 60° C., and has a specific gravity of 0.69 to 0.72 at 15.5° C.

(3) Pure Benzine smells of petroleum.

(4) It dissolves iodine, the solution being raspberry-red.

(5) If brought in contact with coal-tar pitch, even for a long time, it dissolves very little of the latter and becomes scarcely colored.

(6) When shaken in the cold with one-third of its volume of melted crystals of pure carbolic acid, the latter is not dissolved, but forms a layer by itself separate from the Benzine.

(7) Pure Benzine requires for a complete solution at an ordinary temperature 2 volumes of absolute alcohol, or 4 to 5 volumes of methyl alcohol of 0.828 specific gravity.

(8) Heated with 4 volumes of nitric acid (Reagent 15) of 1.45 specific gravity, the latter becomes brown, while the Benzine is but little attacked and forms an upper layer.

### Test for the Purity of Bergamot-oil.

(1) Pure Oil of Bergamot should dissolve at 20° C. in 1½ to 2 volumes of 80% alcohol. Slight turbidity, increasing on the addition of more alcohol, is due to separation of bergaptene, but none of the oil should remain undissolved, and the specific gravity at 15° C. should not be under 0.881.

### Test for the Purity of Black Pepper.

(1) A portion of the powdered Pepper is extracted by maceration or percolation with ether. To the ethereal solution a little water is added, followed by a few drops of ammonia. On shaking, a deep lilac-red color is obtained in the aqueous layer if adulterated with *Myrsine africana* or *Embelia ribes*. Pure Pepper gives no such color.

### Test for the Purity of Borax, $\text{Na}_2\text{B}_4\text{O}_7 + 10\text{H}_2\text{O}$ .

Borax occurs in colorless, transparent, prismatic crystals, or as a white powder; it is odorless, and has a sweetish, alkaline taste. In warm, dry air it is slightly efflorescent. It is soluble in 20.4 parts of water at 25° C., in 0.5 part of boiling water and 1 part of glycerin at 80° C.; insoluble in alcohol. When heated, Borax at first loses part of its water of crystallization, then melts, and, when further heated, swells up and forms a white, porous mass. At a red heat it loses all of its water of crystallization (47 per cent), and fuses to a colorless glass. To a non-luminous flame it imparts an intense yellow color. An aqueous solution (1 in 20) colors red litmus paper blue, and yellow turmeric paper reddish brown.

(1) If a drop of the solution of Borax in glycerin be

held in a non-luminous flame, a transient bright-green color will appear.

(2) If a slight excess of diluted sulfuric acid be added to a hot, saturated, aqueous solution of Borax, shining, scaly crystals of boric acid will separate on cooling, which, when dissolved in alcohol and the liquid ignited, imparts a green color to the flame.

(3) With 21 c.c. (340 minims) of water 1 gm. ( $15\frac{1}{2}$  grs.) of Borax should yield a perfectly clear and colorless solution, leaving no residue.

(4) The aqueous solution of Borax (1 in 20) should not effervesce with acids (absence of *carbonate* or *bicarbonate*).

(5) The aqueous solution of Borax (1 in 20) should not be rendered turbid by magnesia mixture (absence of *phosphate*).

(6) If 1 gm. ( $15\frac{1}{2}$  grs.) of Borax be dissolved in 20 c.c. (324 minims) of diluted sulfuric acid by the aid of heat, and 3 drops of indigo T.S. be added, the blue color should not be discharged after heating for ten minutes on a water-bath (absence of *nitrate*).

### Test for the Purity of Butter.

(1) Heat the suspected Butter in a crucible or test-tube to about  $148.8^{\circ}$  to  $160^{\circ}$  C. At this temperature artificial Butter froths but little, and the mass exhibits irregular movements resembling those of boiling, accompanied by sudden, forcible shocks which frequently throw a part of the fat from the crucible. Casein at the same time is separated and forms on the edge of the crucible in small balls, which assume a brown tint, while the fat retains its original color. Genuine Butter, under these circumstances, foams copiously, the agitation occasioned by

boiling is not nearly so forcible, and the entire mass assumes a uniform brown color.

(2) Melt and filter the suspected Butter. Then take 10 grs. of this, heat it in a test-tube to  $66^{\circ}$  C., then add 30 minims of phenol, shake the mixture and heat it in a water-bath until the fluid becomes transparent. The test-tube is then allowed to stand quietly for some time. Genuine Butter will give a clear solution, but suet, tallow, or lard forms two separate layers of fluid, the upper of which becomes turbid on cooling.

(3) Stir half a teaspoonful or less of the suspected Butter in enough sulfuric ether to dissolve it. By the time the grease is dissolved the ether will have been evaporated and the residuum will show, to smell or taste, whether it is Butter, lard, or tallow.

(4) Heat some of the sample in a spoon over a naked flame until it boils. Pure Butter melts, foams, and runs over the spoon, with an odor of Butter only; oleomargarin melts with much spluttering, foams slightly, and the odor of hot beef-fat is strongly marked. Continue the heat until the sample ignites, and compare the odor given off with that of a sample known to be pure Butter similarly treated.

(5) Heat 1 gm. ( $15\frac{1}{2}$  grs.) of sample with about 5 c.c. (81 minims) of a 10 per cent alcoholic solution of caustic potash until the fat is saponified, then pour into cold water. Pure Butter gives off a marked odor of pineapples (from butyric ether); oleomargarin does not give any marked odor.

(6) Put about as much as a big hickory nut of the sample into a beaker nearly filled with fresh milk, heat gently until the fat melts and spreads over the surface of the milk, now remove heat, and as the milk cools stir the fat continuously with a splinter of wood until the fat congeals. At congealing point Butter will granulate

and cannot be collected in one mass; oleomargarin can easily be collected in one lump. The distinction is very marked, and if the test be tried on a sample of pure Butter first, there will be no difficulty in making the distinction. Lard, cottolene, and Butter with 75 per cent or more oleomargarin will behave like oleomargarin.

(7) Pour the melted sample on fullers' earth—azocolors will color the mass red, the coloring being soluble in alcohol, giving a yellow color with sulfuric acid, becoming pink when diluted with water.

(8) Shake 5 gms. (77 grs.) of the sample with 2 c.c. (32 minims) carbon disulfid and 15 c.c. ( $\frac{1}{2}$  f. oz.) alcohol—the color will be dissolved by the alcohol, which can be evaporated and the residue tested. A green-blue color with sulfuric acid indicates annatto; a brown color indicates boric acid.

(9) Shake a small quantity of the Butter in alcohol and set aside for two or three minutes; then decant the alcohol and evaporate it over a spirit-lamp. There should be no non-volatilizable residue, the Butter should yield nothing to the alcohol. In case the Butter is colored with annatto, a brownish-red sediment forms at the bottom of the dish, which is changed to a blue color by the addition of sulfuric acid (Reagent 22). If it be colored with curcumin, a deep rose-colored residue will remain which turns brown when treated with hydrochloric acid (Reagent 5), and an intense brown when treated with potassium carbonate or sodium carbonate. Saffron produces a sediment which becomes red when treated with lead subacetate, and carrots produce one that turns green with alkali.

(10) Place a heaping teaspoonful of the sample in a teacup, add a couple of teaspoonfuls of hot water, and stand the cup in a vessel containing a little hot water until the Butter is thoroughly melted. Mix the contents

of the cup well by stirring with a teaspoon and set the cup with the spoon in it in a cold place until the Butter is solid. The spoon with the Butter (which adheres to it) is now removed from the cup and the turbid liquid remaining strained through a white cotton cloth, or better, through filter-paper. The liquid will not all pass through the cloth or filter-paper, but a sufficient amount for the test may be secured readily. About a teaspoonful of the liquid is placed in any dish not metal, and 5 drops of hydrochloric acid (Reagent 5) added. A strip of turmeric paper is now dipped into the liquid and then held in a warm place—near a stove or lamp—till dry. If boric acid or borax was present in the sample, the turmeric paper becomes bright cherry-red when dry. A drop of household ammonia changes the red color to dark green or greenish black.

### Test for the Purity of Calomel, $\text{HgCl}$ .

Calomel is a white, impalpable powder, becoming yellowish white on being triturated with strong pressure; it is odorless and tasteless and permanent in the air; insoluble in water, alcohol, or ether, and also in cold dilute acids. When strongly heated it is volatilized without fusion or the evolution of brown vapors, leaving no appreciable residue.

(1) Calomel is blackened in contact with calcium hydroxid T.S., or with solutions of alkali hydroxids, or with ammonia-water (Reagent 1).

(2) When Calomel is heated with dried sodium carbonate in a dry test-tube, it yields a sublimate of metallic mercury.

(3) If 1 gm. ( $15\frac{1}{2}$  grs.) of Calomel be shaken with 10 c.c. (162 minims) of water or alcohol, and the mixture filtered, neither of the filtrates should respond to

the Time-limit Test for *heavy metals* (see Test for the Purity of Glycerin (7)), nor should any appreciable residue be left on evaporation (absence of *soluble impurities*).

(4) If 2 gm. (31 grs.) of Calomel be shaken with 20 c.c. (324 minims) of ether, filtered, the filtrate evaporated, and 10 c.c. (162 minims) of distilled water added, not more than a slight opalescence should result upon the addition of silver nitrate T.S. to 5 c.c. (81 minims) of the filtrate, and no change in color should be produced upon adding a few drops of ammonium sulfid T.S. to the remainder (absence of *mercuric chlorid*).

(5) On heating a portion of Calomel in a test-tube with potassium hydroxid T.S., it should not evolve ammonia; and if another portion be shaken with acetic acid and filtered, the filtrate should not be affected by hydrogen sulfid T.S. nor by silver nitrate T.S. (distinction from and absence of *ammoniated mercury*).

(6) If to 0.5 gm. (8 grs.) of Calomel contained in a small beaker, 5 c.c. (81 minims) of nitric acid (Reagent 15) be added, and the mixture evaporated to dryness on a water-bath, and if, after dissolving the residue in about 25 c.c. (405 minims) of distilled water and 5 c.c. (81 minims) of hydrochloric acid (Reagent 5), the solution be completely saturated with hydrogen sulfid, and allowed to stand for several hours in a well-corked flask until the precipitate has subsided and then filtered, the filtrate should be colorless, and leave no weighable residue upon evaporation (absence of many *foreign salts*).

(7) If the precipitate obtained in the preceding test (6), after washing with about 1000 c.c. (3 f. oz., 183 minims) of water and draining, be rinsed into a beaker with about 20 c.c. (324 minims) of water and then 5 c.c. (81 minims) of stronger ammonia-water added, and if, after covering and digesting the mixture for about fifteen

minutes on a water-bath, it be rinsed upon a filter and washed with a little water, the filtrate and washings, after evaporating to dryness, moistening with 6 drops of nitric acid (Reagent 15), and again drying, should not respond to the Test for Arsenic (see Test for Arsenic (8)).

### **Test for Purity of Canned Peas, Beans, Spinach, etc.**

(1) Mash some of the sample in a dish with a stiff kitchen-spoon. Place a teaspoonful of the pulp in a tea-cup with three teaspoonfuls of water and add 30 drops of strong hydrochloric acid (Reagent 5) with a medicine-dropper. Set the cup on the stove in a saucepan containing boiling water. Drop a bright iron brad or nail (wire nails are the best, tin carpet-tacks will not answer the purpose) into the cup and keep the water in the saucepan boiling for twenty minutes, stirring the contents of the cup frequently with a splinter of wood. Pour out the contents of the cup and examine the nail. If the sample has been adulterated with bluestone, the nail will be plated with copper.

### **Test for the Purity of Castor-oil.**

Castor-oil is a pale yellowish or almost colorless, transparent, viscid liquid, having a faint, mild odor, and a bland, afterwards slightly acrid and generally offensive taste. Specific gravity: 0.945 to 0.965 at 25° C. Soluble in an equal volume of alcohol, and in all proportions in absolute alcohol or in glacial acetic acid; also soluble at 25° C. in 3 times its volume of 92.5 per cent alcohol (absence of more than about 5 per cent of *most other fixed oils*). With an equal volume of petroleum benzin it forms at 15° C. a turbid mixture, but at 17° C. it yields

a clear solution. When cooled to  $0^{\circ}\text{C}$ . it becomes turbid, with the separation of crystalline flakes, and at about  $-18^{\circ}\text{C}$ . it congeals to a yellowish mass.

(1) If 3 c.c. (48 minims) of the oil be shaken for a few minutes with 3 c.c. (48 minims) of carbon disulfid and 1 c.c. (16 minims) of sulfuric acid (Reagent 21), the mixture should not acquire a blackish-brown color (absence of *foreign oils*).

(2) Cudbear when boiled in alcohol, filtered, evaporated to dryness and heated with pure Castor-oil exhibits a brilliant orange, fluorescent light. Camwood yields an apple-green, while turmeric yields a vivid emerald-green.

(3) If 1 volume of Castor-oil is agitated with 2 volumes of benzin and then set aside, two layers are formed after several hours, standing, the lower one being increased at  $16^{\circ}\text{C}$ . to one-sixth of the original volume, while at  $10^{\circ}\text{C}$ . it is increased to 1.75. A certain portion of the benzin is dissolved by the Castor-oil, while a much smaller portion (about one-thirtieth) of the Castor-oil dissolves in the benzin. If the Castor-oil, however, is adulterated with other fixed oils, a clear solution may form, or, if two layers are formed, the lower layer will be less than 1.5 times the original volume. Taking the increase in volume when operating with pure Castor-oil at 1.5, if the oil is adulterated to the amount of 10 per cent, the volume will be 1.0 to 1.2; if 25 per cent, it will be only about 0.4

(4) Castor-oil is specifically one of the heaviest fixed oils, and when dissolved in alcohol is rendered lighter, so that any foreign oil with which it may be adulterated separates to the bottom.

(5) Mix 1 volume of the Castor-oil to be tested with 2 volumes of alcohol of exactly 0.838 specific gravity, heat, and stir well with a thermometer till complete solution. In the case of pure Castor-oil this will be be-

tween 38° and 43° C., possibly lower than the former; whilst if any foreign oil be present, the temperature will be much higher, and in gross adulteration some oil may not be dissolved even at the boiling-point of the mixture.

(6) Castor-oil treated with silver nitrate should not give a red color (absence of *cottonseed-oil*).

(7) Rosin-oil may be detected in Castor-oil by saponification and agitation of the aqueous soap solution with ether, separating and evaporating the ethereal solution, when the rosin-oil will be obtained.

(8) Pure Castor-oil is colored only slightly brown with nitric acid of specific gravity 1.31, while the acid solution remains colorless; in the presence of rosin-oil the oily layer will be decidedly dark-colored (almost black) and the acid layer will be of a brownish color.

(9) Heat the Castor-oil in a small porcelain dish; a distinctive odor of cocoanut-oil indicates its presence as an adulterant.

(10) Make a test solution containing 5 parts of silver nitrate and 1 part of nitric acid (sp. gr. 1.42) in one hundred parts of alcohol (sp. gr. 0.838). Pour about 6.5 gm. (100 grs.) of the oil to be examined into a dry test-tube about half an inch in diameter, add to it .648 gm. (10 grs.) of the above test solution, and place the test-tube in boiling water for five minutes. Pure Castor-oil assumes a pale-yellow color, but the presence of cottonseed-oil causes it to become deep red.

### Test for the Purity of Chloroform, $\text{CHCl}_3$ .

Chloroform is prepared by heating, over a water-bath, ethyl alcohol ( $\text{C}_2\text{H}_5\text{OH}$ ) with bleaching-powder ( $\text{CaOCl}_2$ ) in a large flask fitted to a condenser. It is a heavy, clear, colorless, mobile, and diffusible liquid, of a characteristic ethereal odor, and a burning, sweet taste. Specific

gravity not below 1.476 at 25° C. It is soluble in about 200 times its volume of cold water, and in all proportions in alcohol, ether, benzin, petroleum benzin, and in the fixed and volatile oils. Chloroform is volatile even at a low temperature, and boils at 60° to 61° C. It is not inflammable, but its heated vapor burns with a green flame. It is neutral to litmus paper.

(1) If 10 c.c. (162 minims) of Chloroform be poured upon a clean, odorless filter-paper laid flat upon a warmed glass plate, and the plate be rocked from side to side until the liquid is all evaporated, no foreign odor should become perceptible as the last portions disappear from the paper, and the paper should be left odorless.

(2) If 10 c.c. (162 minims) of Chloroform be well shaken with 20 c.c. (324 minims) of distilled water, and the liquid be allowed to separate completely, the water should be neutral to litmus paper, and should not be affected by silver nitrate T.S. (absence of *chlorides*), nor colored by potassium iodid T.S. (absence of *free chlorin*).

(3) If 40 c.c. (1 f. oz., 169 minims) of Chloroform be shaken with 4 c.c. (64 minims) of colorless, concentrated sulfuric acid (Reagent 21) in a 50 c.c. (1 f. oz., 331 minims) glass-stoppered cylinder during twenty minutes, and the liquids be then allowed to separate completely, so that both are transparent, the Chloroform should remain colorless, and the acid should appear colorless or very nearly colorless, when seen in a stratum of not less than 15 mm. ( $\frac{5.9}{800}$  inch) in thickness (absence of *impurities decomposable by sulfuric acid*).

(4) If 2 c.c. (32 minims) of the sulfuric acid, separated from the Chloroform, be diluted with 5 c.c. (81 minims) of distilled water, the liquid should be colorless and clear, and, while hot from the mixing, should be odorless, or give but a faint vinous or ethereal odor (absence of *odorous decomposition products*). When fur-

ther diluted with 10 c.c. (162 minims) of distilled water, it should remain clear, and should not be affected by silver nitrate T.S. (absence of *chlorinated decomposition compounds*).

(5) Chloroform, when boiled with potassium-hydrate solution containing a fragment of resorcin, gives an intense red color (rosolic acid).

(6) Add a drop of anilin to an alcoholic solution of potassium hydrate, then add 1 or 2 drops of the suspected liquid and boil, a fearfully offensive odor of phenylisocyanid is produced if Chloroform is present.

(7) When 0.5 c.c. (8 minims) of a 5 per cent alcoholic solution of thymol is treated with a drop of Chloroform and a fragment of caustic potash and boiled, the mixture becomes yellow, then red; on adding 1 c.c. (16 minims) of sulfuric acid (Reagent 21) to this red mixture and again heating, a very intense violet color is obtained.

#### **Test for the Purity of Cocoanut-oil.**

Pure Cocoanut-oil is of a fine white color, of the consistency of lard at ordinary temperatures, becoming solid, like suet, between  $4.4^{\circ}$  and  $10^{\circ}$  C. and liquid at about  $26.6^{\circ}$  C.; it has a bland taste and a peculiar, not disagreeable, odor. It is readily dissolved by alcohol.

#### **Test for the Purity of Cod-liver Oil.**

Cod-liver Oil is a pale yellow, thin, oily liquid, having a peculiar, slightly fishy, but not rancid odor, and a bland, fishy taste. Specific gravity 0.918 to 0.922 at  $25^{\circ}$  C. Very slightly soluble in alcohol, but readily soluble in ether, chloroform, or carbon disulfid; also in 2.5 parts of acetic ether.

(1) If 1 drop of the Oil be dissolved in 20 drops of chloroform and the solution shaken with 1 drop of sul-

furic acid (Reagent 21), the solution will acquire a violet-red tint, rapidly changing to rose-red and finally to brownish yellow.

(2) If a glass rod moistened with sulfuric acid (Reagent 21) be drawn through a few drops of the Oil on a porcelain plate, a violet color will be produced.

(3) Cod-liver Oil should be only very slightly acid to blue litmus paper, which has been previously moistened with alcohol (limit of free fatty acids).

(4) If 2 or 3 drops of fuming nitric acid be allowed to flow alongside of 10 or 15 drops of the Oil contained in a watch-glass, a red color will be produced at the point of contact. On stirring the mixture with a glass rod, this color becomes bright rose-red, soon changing to lemon-yellow (distinction from *seal-oil*, which shows at first no change of color, and from *other fish-oils*, which become at first blue and afterwards brown and yellow).

### Test for the Purity of Coffee.

(1) Add basic acetate of lead to the decoction, filter, and precipitate the excess of lead by means of sulfid of hydrogen. When evaporating the clear remaining liquid you may crystallize out the caffenin, if the decoction contained real Coffee.

(2) Pour freshly made chlorin-water on the remnant of a dried-up solution of the Coffee to be tested, and this, dried again, will become red as blood, if real Coffee is present. This test is very delicate.

### Test for the Purity of Copaiva.

(1) Mix 1 gm. (9 minims) of pure concentrated sulfuric acid (Reagent 22) with 25 gms. (7 f. drs.) of absolute acetic ether. When to 1 to 2 c.c. (16 to 32 minims) of

this solution 2 to 4 drops of the Copaiva are added, there should appear no red on violet coloration.

(2) If 1 volume of the Copaiva be thoroughly shaken with 3 or 4 volumes of water, filtered through a wetted filter and treated with an equal volume of hydrochloric acid sp. gr. 1.120, no pink coloration should occur within fifteen minutes.

### **Test for the Purity of Cream of Tartar.**

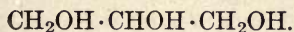
(1) As found in commerce Cream of Tartar usually contains from 2 to 5 per cent of tartrate of lime; and hence a little carbonate of lime may be detected in black flux. To detect the tartrate of lime digest the suspected Cream of Tartar with a solution of caustic ammonia, and test the filtered solution with ammonium oxalate. A white precipitate indicates lime.

(2) If the Cream of Tartar be adulterated with either alum or potassium bisulfate, the fraud may be detected by barium chlorid, which causes a white precipitate (sulfate of baryta) insoluble in nitric acid.

(3) Sulfureted hydrogen and solution of potassium ferrocyanid should produce no change in a solution of Cream of Tartar.

(4) Saturate a solution of Cream of Tartar with pure potassium carbonate, then add lime-water, and afterwards sal ammoniac. If the sal ammoniac does not completely dissolve, the precipitate caused by the lime-water, racemate of lime, is present. This is the test for racemic acid.

(5) Other adulterants of Cream of Tartar are gypsum, chalk, alum, and starch. The gypsum, chalk, and alum may be detected by boiling a solution of the Cream of Tartar with oxalate of barium; the starch by adding iodine to a cold solution.

**Test for the Purity of Glycerin. Glycerol.**

Glycerin is a liquid obtained by the decomposition of vegetable or animal fats or fixed oils. It is a clear colorless liquid, of a thick, syrupy consistence, smooth to the touch, odorless, sweet to the taste, and producing a sensation of warmth in the mouth; when exposed to the air it absorbs moisture. Specific gravity not less than 1.250 at 15° C.; boils at 165° C. It is soluble in all proportions in water and alcohol, also soluble in a mixture of 3 parts of alcohol and 1 part of ether, but insoluble in ether, chloroform, carbon disulfid, petroleum benzin, benzene, and fixed and volatile oils. An aqueous solution of Glycerin is neutral to litmus paper.

(1) If a fused bead of borax on a loop of platinum wire be moistened with Glycerin and then held in the edge of a non-luminous flame, the latter will be transiently tinted a vivid green.

(2) 5 c.c. (81 minims) of Glycerin heated to boiling in an open porcelain or platinum dish and then gently ignited should vaporize, burn, and leave not more than a dark stain, which on stronger heating should disappear entirely (absence of *mineral impurities*).

(3) If 5 c.c. (81 minims) of Glycerin be mixed with 50 c.c. (1 f. oz., 331 minims) of water and 10 drops of hydrochloric acid (Reagent 5) in a small flask and heated for half an hour on a water-bath, then 10 c.c. (162 minims) of the hot liquid, mixed with 2 c.c. (32 minims) of sodium hydroxid T.S. and 1 c.c. (16 minims) of alkaline cupric tartrate V.S., should show no yellowish-red cloudiness or precipitate within six hours (absence of *sugars*).

(4) If 5 c.c. (81 minims) of Glycerin be mixed with an equal volume of concentrated sulfuric acid (Reagent 21) in a test-tube, the liquid should acquire, on standing for

one hour, a color not darker than yellow (absence of *readily carbonizable impurities*).

(5) If 5 c.c. (81 minims) of Glycerin be mixed with the same volume of a mixture of equal parts of alcohol and diluted sulfuric acid and gently heated, a fruity odor should not be recognizable (absence of *butyric acid*).

(6) No color, cloudiness, or precipitate should appear when separate portions of an aqueous solution of Glycerin (1 in 10) are treated with barium chloride T.S. (*sulfuric acid*), calcium chloride T.S. (*oxalic acid*), ammonium oxalate T.S. (*calcium salts*), silver nitrate T.S. (*chlorides*), or silver ammonium nitrate T.S. (*acrolein*); in the last-mentioned case the test-tube, loosely stoppered to protect it from impurities, should be allowed to stand, protected from light, for at least five minutes.

(7) 10 c.c. (162 minims) of a solution of Glycerin in distilled water (1 in 20), contained in a test-tube of about 40 c.c. (1 f. oz., 331 minims) capacity, is acidulated with 1 c.c. (16 minims) of diluted hydrochloric acid, warmed to about 50° C., and an equal volume of freshly prepared hydrogen sulfid T.S. added, and the mixture allowed to stand, in the well-stoppered test-tube, in a warm place, at 35° C. for at least half an hour. At the end of this time any coloration or turbidity is carefully noted; *antimony* yields a pale-yellow to orange color or orange precipitate; *arsenic* yields a pale-yellow color or yellow precipitate; *copper* yields a pale-brown color or a brownish-black precipitate; *lead* yields a pale-brown color or a black precipitate; *zinc* yields a white turbidity or white precipitate.

(8) 5 c.c. (81 minims) of the aqueous solution of Glycerin (1 in 10) should not respond to the test for *arsenic* (see Test for Arsenic (8)).

(9) Pure Glycerin when rubbed between the fingers does not emit a fatty smell.

### Test for the Purity of Gold-leaf.

(1) Put a few drops of nitric acid (Reagent 15) upon a piece of glass and place thereon a small piece of the Gold-leaf. Pure Gold-leaf will not be affected by the acid, while other metals that may be mixed with it will go into solution.

(2) Gold should dissolve in a mixture of one part of nitric acid with three parts of hydrochloric acid. A residue indicates silver. If sulfuric acid is added to the solution, a precipitate indicates lead. One quick method is to determine its specific gravity.

### Test for the Purity of Gum Shellac.

(1) Take ten parts by weight of the suspected article, five parts of powdered borax, and 400 parts by weight of water, boil until a complete solution is had, then add enough water to throw down all impurities. Collect the precipitate upon a cloth filter, wash it thoroughly and dry. The dry precipitate must not amount to over five per cent by weight of the amount of Shellac used and if the solution is cloudy and opalescent, it may be taken for granted that the Gum Shellac has been adulterated with ordinary rosin or crude turpentine.

### Test for the Purity of Honey.

Honey is a syrupy liquid of a light-yellowish to yellowish-brown color, translucent when fresh, but gradually becoming opaque and crystalline, having a characteristic, aromatic odor and a sweet, faintly acrid taste.

When recent Honey is diluted with twice its weight of water, the resulting liquid should be almost clear, not stringy, and should have a specific gravity not lower than

1.099 (corresponding to a specific gravity of 1.370 for the original Honey). When Honey is incinerated in small portions at a time in a platinum crucible, it should not leave more than 0.3 per cent of ash. Honey has a faintly acid reaction, due to formic acid, upon litmus paper, and is lævogyrate.

(1) If 5 gm. (77 grs.) of Honey be dissolved in 20 gm. (308 grs.) of water, a clear or nearly clear solution will result, which should not be rendered more than faintly opalescent by a drop of silver nitrate T.S. (limit of *chlorides*), or of barium chloride T.S. (limit of *sulfates*).

(2) If 2 c.c. (32 minims) of a filtered solution of the Honey (1 in 4) be placed in a test-tube 1 cm. ( $\frac{3.9}{100}$  inch) in diameter, and 1 c.c. (16 minims) of absolute alcohol be allowed to flow down the walls of the tube held in an inclined position, so as to form an overlying layer, this should remain clear, and the line of contact should not show more than barely noticeable opalescence, which soon disappears; a permanent milky zone should not be produced (absence of *starch sugar*).

(3) If 2 c.c. (32 minims) of pure concentrated sulfuric acid (Reagent 21) be placed in a test-tube of 1 cm. ( $\frac{3.9}{100}$  inch) diameter, and 0.5 c.c. (8 minims) of a solution of Honey (1 in 4) be allowed to flow upon it so as to form a distinct upper layer, no colored line of contact should show at once, and at the end of one hour the zone of contact should be at most yellowish or clear brown; a brownish color becoming nearly black at the end of half an hour should not develop (absence of *cane-sugar*).

(4) On boiling 1 part of Honey with 5 parts of water, the resulting solution, when cold, should not be rendered blue or green on the addition of iodine T.S. (absence of *starch*).

(5) Gum and gelatin are detected by dissolving the Honey in a little water, and evaporating to a syrupy

consistence, when these adulterations will cause the mass to gelatinize. Gelatin can also be directly precipitated by tannic acid. Heating with quicklime in a test-tube will cause vapor of ammonia to be given off if gelatin is present.

### **Test for the Purity of Jam, Jelly, Marmalade, and Sausage.**

(1) Macerate the food with water and strain through a piece of white cotton cloth. The maceration may be performed by rubbing in a teacup or other convenient vessel with a heavy spoon. Between 60 and 100 c.c. (2 and 3 f. oz.) of the liquid obtained from the food, as described above, are placed in a narrow bottle holding 150 c.c. (5 f. oz.), about a quarter of a teaspoonful of cream of tartar (or, better, a few drops of sulfuric acid) is added, the mixture shaken for two or three minutes, and filtered into a second small bottle. Three or four tablespoonfuls of chloroform are added to the clear liquid in the second bottle, and the liquids mixed by a somewhat vigorous rotary motion, poured into an ordinary glass tumbler, and allowed to stand till the chloroform settles out in the bottom. Shaking is avoided, as it causes an emulsion which is difficult to break up. As much as possible of the chloroform layer is removed (without any admixture of the aqueous liquid) by means of a medicine dropper and placed in a test-tube with about an equal amount of water and a small fragment—a little larger than a pinhead—of iron alum. The mixture is thoroughly shaken and allowed to stand till the chloroform again settles to the bottom. The presence of salicylic acid is indicated by the purple color of the upper layer of liquid.

(2) Extract the sample with chloroform as in (1); remove the chloroform layer and place it in a white saucer,

or, better, in a plain glass sauce-dish. Set a basin of water—as warm as the hand can bear—on the outside window ledge and place the dish containing the chloroform extract in it, closing the window until the chloroform has completely evaporated. Benzoic acid, if present in considerable amount, will now appear in the dish in characteristic flat crystals. On warming the dish the unmistakable irritating odor of benzoic acid may be obtained. This method will detect benzoic acid in tomato catsup or other articles in which it is used in large quantities.

(3) If the sample contains insoluble material, as sausage, it may be treated with sufficient water to dissolve the soluble portion with the formation of a thin liquid and filtered, and then strained through a clean white cotton cloth to separate the insoluble portion. About a half teacupful of the liquid is heated to boiling, after adding a few drops of hydrochloric acid (Reagent 5) and a small piece of white woolen cloth or a few strands of white woolen yarn. (Before using, the wool should be boiled with water containing a little soda, to remove any fat it may contain, and then washed with water.) The wool is again washed, first with hot and then with cold water, the water pressed out as completely as possible, and the color of the fabric noted. If no marked color is produced, the test may be discontinued and the sample considered free from artificial colors. If the fabric is colored, rinse it in hot water, and then boil for two or three minutes in about one-third of a teacupful of water and two or three teaspoonfuls of household ammonia. It turns purple or blue if the sample has been artificially colored with an anilin dye.

#### **Test for the Purity of Lactic Acid.**

(1) Take 1 volume of the suspected Acid and add about 10 volumes of ether; this serves to throw out of

solution glycerin or syrup, because these are immiscible with ether. If it contains these, the mixture will have a milk-like color, and the syrup or glycerin will settle to the bottom on standing. The upper layer will consist of the ether and Lactic Acid, and the lower layer of either glycerin or syrup. Now separate the lower layer by means of a separating-funnel or pipette, place it in a crucible, bring it to the boiling-point, and ignite; if it chars, it shows the presence of sugar, and if it burns and leaves a black stain it shows the presence of glycerin.

(2) On adding a few drops of Lactic Acid to hot Fehling's solution no red cuprous oxide should be separated (absence of sugar).

(3) If a small portion of the acid be heated with an excess of zinc carbonate, the mixture dried at  $100^{\circ}\text{C.}$ , and then extracted with absolute alcohol, upon evaporation of the latter no sweet residue should remain (absence of glycerin).

### Test for the Purity of Lampblack.

Lampblack is a smoke produced by burning (with a limited supply of oxygen) oils, gases, etc., rich in carbon. The smoke thus produced, by means of a sluggish draught, is conducted into condensing chambers, in which, after some hours, the particles which are in an exceedingly finely divided state deposit. True Lampblack, therefore, must be a smoke and is not a residue or charcoal, consequently, no air-floated pigment is or can be in a more finely divided state than true Lampblack.

Most of the substitutes and the materials used in adulterating Lampblack are mineral or shale blacks, wood charcoal, coal, and coke-dust, chemical residues, by-products, etc. Some of these are blacker than pure Lampblack, but all lack its marked characteristics.

The simple tests here given will enable purchasers and users of black pigments to detect spurious substitutes branded Lampblack from true Lampblack; also the adulteration in Lampblack, besides determining the relative value of two Lampblacks as to color, covering, spreading, tinctorial power, etc.

(1) The best test of permanency is time and actual wear and tear, but the following will be of assistance in estimating durability: Upon the dry pigment pour a few drops of sulfuric acid (Reagent 21); if an effervescence occurs, you may be quite sure that the black is not pure Lampblack, or that some adulterant has been used. If sulfuric acid (Reagent 21) or a strong solution of ammonia or caustic soda (Reagent 20) makes the color bleed or run, and, when dried, the pigment has been robbed of color, you may be satisfied the color is fugitive and not durable.

(2) Take the samples of black pigment and mix in grain-alcohol, then, with a camel's-hair brush, spread upon a slip of glass; when the blacks are so spread, hold the glass before a strong light. Pure Lampblack will be found to perfectly cover the glass and be impervious to light, while the adulterated black or spurious black will not be opaque, for the particles forming the pigment, not being in a finely divided state, will not form a dense black mass.

(3) An absolute test for purity and detection of an adulterant in Lampblack is obtained by burning out the black in a porcelain crucible over the gas-flame of a Bunsen burner. Coke, residue, etc., or any impurity or adulteration of Lampblack will remain in the crucible in the form of ash; that black which contains the largest percentage of ash will be the least permanent in color, and paint made with it will not go so far nor last as long as that made with a pure Lampblack which contains absolutely no ash.

(4) Upon a piece of white blotting-paper put a few grains of Lampblack, and slowly pour on sufficient sulphuric ether to wet the blotting-paper beyond the surface covered by the black. If the black contains any free oil, the paper around the black disk will be discolored a brownish yellow.

### Test for the Purity of Lard.

(1) Pure Lard melted and mixed with strong sulphuric acid (Reagent 21) or nitric acid (Reagent 15) will give only a slight color, which may be yellowish, pinkish, or inclined to light brownish. Cottonseed-oil and other seed-oils and mixtures containing them, similarly treated, yield any color between yellowish brown and very brownish black or even black.

(2) With nitrate of silver solution pure Lard causes no more than the very slightest amount of reduction, and generally none at all, but cottonseed-oil causes a very marked reduction of the salt to the metallic state, with the result that the mixture has a brownish or black appearance from the minute black particles formed.

### Test for the Purity of Linseed-oil.

Linseed-oil, if pure, should weigh very closely to  $7\frac{3}{4}$  pounds per gallon of 231 cubic inches in the crude or raw state, and a good fire or kettle-boiled Linseed-oil about 7 pounds 13 ounces to 7 pounds 14 ounces per gallon. The commercial custom, however, is to sell Linseed-oil by the gallon, taking it at  $7\frac{1}{2}$  pounds.

(1) Pure Linseed-oil placed on the tongue has a peculiar bland taste, finally creating a slight irritation. If mixed with fish-oil, even in small proportions, the taste becomes nauseating; with mineral-oil or rosin-oil the taste is such that the sensation will adhere to the tongue

for a day or two. If mixed with corn-oil, the taste is nearly sweet and rather pleasant. With cottonseed-oil it is somewhat like lard, though not as pleasant.

(2) Pure raw Linseed-oil when briskly rubbed between the palms of the hands must heat quickly, give a sort of whitish lather, and emit the characteristic odor of linseed-meal. When adulterated with fish-oil, the odor is nauseating in the extreme, no matter how much the fish-oil in the mixture has been deodorized. With rosin-oil or mineral-oil the odor is certain to reveal the characteristics of either of these on the hands becoming warmed up by friction.

With corn-oil the mealy odor cannot be mistaken, and with cottonseed-oil the lard-like smell will be apparent.

(3) The presence of mineral-oil or rosin-oil in Linseed-oil, even in the so-called debloomed grades, can be readily detected by dropping a small portion of the suspected oil on a strip of glass that has been painted with a strong black paint, preferably lampblack, not too glassy, and alongside of it a similar portion of pure raw Linseed-oil. No matter how small the percentage of adulteration may be, the sophisticated article will show a decidedly blue-gray color which in the case of mineral-oil, in strong sunlight especially, will be very marked.

(4) Equal parts of the suspected oil and nitric acid, 1.40 sp. gr., are placed into a test-tube and shaken very thoroughly, then stood aside until two strata have formed in the tube on the separation of the oil and acid, which will take place in from ten to fifteen minutes, and the color of the two strata is observed. When the oil is pure linseed, the upper stratum is a light cinnamon-brown, the lower colorless in the case of raw oil; while in boiled oil the upper stratum may be a trifle darker, but the lower must also be colorless. When adulterated with rosin-oil, the upper stratum will be found dark olive to

black, and the lower from straw to orange, according to the percentage of rosin-oil that may be present. When fish-oil is present, the upper stratum will vary from brown to a brown-black, the lower one from a light to a dark-orange shade, according to the quantity of fish-oil. When Linseed-oil is mixed with cottonseed-oil, the upper stratum will turn reddish brown, the lower one very pale yellow, more or less so, according to the quantity of cottonseed-oil present.

(5) Shake the suspected oil with concentrated solution of potash or soda containing a little grain-alcohol, and then add warm water and shake again. Allow to stand for half an hour, and if any petroleum (paraffin-oil) is present it will separate from the soap and float on top.

(6) Pure raw Linseed-oil has a straw-yellow color, boils at  $130^{\circ}\text{C.}$ , and congeals at  $-27.5^{\circ}\text{C.}$ ; its specific gravity is 0.930 to 0.935.

### **Test for the Purity of Menthol.**

(1) The Menthol is exhausted several times with chloroform until the latter no longer takes up any more of it; the crystals of magnesium sulfate (the usual adulterant) remain behind in their natural state after the chloroform with which they are impregnated has evaporated, and can easily be identified.

### **Test for the Purity of Milk.**

(1) Besides the common method of diluting Milk with water, another species of adulteration has recently been detected. It is now frequently sophisticated with dextrin. This fraud can easily be detected by means of a solution of iodine; if the specimen contains the smallest quantity of dextrin, it will acquire a red color.

(2) A German chemist furnishes a very simple procedure for testing the amount of water in Milk. All that is required is a small quantity of plaster of Paris, say 1 ounce. This is mixed with the Milk to a stiff paste and then allowed to stand. With Milk of 1.030 specific gravity and a temperature of  $15.5^{\circ}\text{C.}$ , it will harden in ten hours; if 25% of water is present, in two hours; if 50%, in one hour and a half; and with 75%, in thirty minutes. Skimmed Milk which has been standing for twenty-four hours and is of 1.033 specific gravity sets in four hours; with 50% of water in one hour; and with 75% in 30 minutes. Heat should not be applied, as then the use of the thermometer would be required.

(3) To 10 c.c. (162 minims) of the Milk to be tested add 1 c.c. (16 minims) of fuchsin sulfurous acid, and allow to stand five minutes; then add 2 c.c. (32 minims) of pure hydrochloric acid (Reagent 5) and shake. If formaldehyde is not present, the mixture remains yellowish white, while, if present, a blue-violet color is produced. This test will detect 0.02 gm. ( $\frac{1}{50}$  gr.) of anhydrous formaldehyde in one litre (34 f. oz.) of Milk.

(4) The reaction of pure fresh Milk is neutral or faintly acid; a Milk of alkaline reaction would suggest the addition of alkaline carbonates or bicarbonates, or borax, as preservatives. The reaction may be taken with good litmus paper, or by floating a solution of litmus on the Milk.

(5) The specific gravity of pure fresh Milk varies from 1.029 to 1.035, and should be taken at  $15.5^{\circ}\text{C.}$ , or reduced to this standard.

(6) Add 1 or 2 drops of a 1 per cent aqueous solution of phenol to a few cubic centimetres of distillate from the Milk and pour the mixture gently on concentrated sulfuric acid (Reagent 21) in a test-tube—a bright-crimson color appears at the point of contact if formaldehyde is present.

(7) Make three solutions: A, Phenylhydrazin hydrochlorate 0.5 per cent in distilled water; B, sodium nitroprussiate 0.5 gm. (8 grs.) in 30 c.c. (1 f. oz.) water; C, liquor sodæ U. S. P. To 15 c.c. ( $\frac{1}{2}$  f. oz.) of Milk add 10 drops of A, mix, add 3 drops of B, put into a test-tube, and let 5 drops of C run down the sides—in the presence of even traces of formaldehyde a blue color is instantly produced, gradually changing on standing to red.

(8) A very simple test is to pour a few cubic centimetres of Milk onto a few grams of powdered potassium sulfate in a flask, and gradually pour in strong sulfuric acid (Reagent 21). If formaldehyde is present, a violet color will appear instead of the brownish black of pure Milk.

(9) Add 0.5 gm. (8 grs.) of powdered ammonium molybdate to 10 c.c. (162 minims) of the Milk, then add 10 c.c. (162 minims) of 10 per cent hydrochloric acid and heat in a test-tube, an intense blue color indicates the presence of cane-sugar.

(10) Formic aldehyde in Milk may be detected by the following method: 100 c.c. (3 f. oz.) of the Milk are carefully distilled until 20 c.c. (324 minims) of distillate are obtained; this is transferred to a stoppered tube and 5 drops of ammonio-nitrate of silver solution added. (This solution is prepared by dissolving 1 gm. ( $15\frac{1}{2}$  grs.) of silver nitrate in 30 c.c. (1 f. oz.) distilled water, adding dilute ammonia-water until the precipitate first formed is redissolved, avoiding an excess; then making up to 50 c.c. (1.69 f. oz.) with distilled water.) The mixture of milk distillate and silver is now allowed to stand several hours (6 to 18) in a dark place, when, if formic aldehyde is present, a strong black color or deposit will be produced. A light-brown color should be disregarded, and heating is useless. Pure Milk when so treated gives no reaction.

(11) 2 or 3 tablespoonfuls of the Milk are placed in a bottle with twice that amount of a solution of a teaspoonful of alum in a pint of water, shaken vigorously, and filtered through filter-paper. About a teaspoonful of the filtrate is placed in any dish, not metal, and 5 drops of hydrochloric acid (Reagent 5) added. A strip of turmeric paper is now dipped into the liquid and then held in a warm place—near a stove or lamp—till dry. If boric acid or borax was present in the sample, the turmeric paper becomes bright cherry-red when dry. A drop of household ammonia changes the red color to dark green or greenish black.

(12) 3 or 4 tablespoonfuls of the sample are placed in a teacup with at least an equal amount of strong hydrochloric acid (Reagent 5) and a piece of ferric alum about as large as a pinhead, the liquids being mixed by a gentle rotary motion. The cup is then placed in a vessel of boiling water, no further heat being applied, and left for five minutes. At the end of this time, if formaldehyde be present, the mixture will be distinctly purple.

### Test for the Purity of Olive-oil.

Olive-oil is a pale yellow or light greenish-yellow oily liquid, having a slight peculiar odor and a nut-like oleaginous taste, with a faintly acrid after-taste. Specific gravity 0.910 to 0.915 at 25° C. It is very sparingly soluble in alcohol, but readily soluble in ether, chloroform, or carbon disulfid. When cooled to from 8° to 10° C. Olive-oil becomes somewhat cloudy from the separation of crystalline particles, and at 0° C. it forms a whitish, granular mass.

(1) If 50 gms. (1 f. oz., 331 minims) of *pure* Olive-oil are placed in a graduated cylinder capable of holding 150 c.c. (5 f. oz., 34 minims), the temperature ascertained,

and 10 c.c. (162 minims) of concentrated sulfuric acid (Reagent 21) gradually added from a pipette and intimately mixed with the oil by stirring for a few minutes, the maximum degree of heat will rise to 42° C.

(2) If 2 c.c. (32 minims) of Olive-oil be shaken vigorously with an equal volume of nitric acid (sp. gr. 1.37), the oil should retain a light-yellow color, not becoming orange or reddish brown, and after standing for six hours should change into a yellowish-white solid mass and an almost colorless liquid (absence of appreciable quantities of *cottonseed-oil* and *most other seed-oils*).

(3) If 5 c.c. (81 minims) of Olive-oil be thoroughly shaken in a test-tube with 5 c.c. (81 minims) of an alcoholic solution of silver nitrate (made by dissolving 0.1 gm. (1½ grs.) of silver nitrate in 10 c.c. (162 minims) of alcohol and adding 2 drops of nitric acid (Reagent 15)), and the mixture be heated for about five minutes in a water-bath, the oil should retain its original pale color, not becoming reddish or brown, nor should any dark color be produced at the line of contact of the two liquids (absence of more than about 5 per cent of *cottonseed-oil*).

(4) If 2 c.c. (32 minims) of Olive-oil be mixed in a test-tube with 2 c.c. (32 minims) of equal volumes of amyl alcohol and carbon disulfid containing 1 per cent of sulfur in solution, and the test-tube be immersed to one-third or one-half its depth in boiling salt water, no reddish color should develop in from ten to fifteen minutes (absence of *cottonseed-oil*).

(5) If 2 c.c. (32 minims) of Olive-oil be mixed with 1 c.c. (16 minims) of hydrochloric acid (sp. gr. 1.18) containing 1 per cent of sugar, and the mixture shaken for half a minute and allowed to stand for five minutes, and then 3 c.c. (48 minims) of water added, and the whole again shaken, the acid layer should not show a pink color (absence of *sesame-oil*).

(6) If 10 c.c. (162 minims) of Olive-oil be shaken frequently during two hours with a freshly prepared solution of 1 gm. ( $15\frac{1}{2}$  grs.) of mercury in 3 c.c. (48 minims) of nitric acid (Reagent 15), a perfectly solid mass of a pale straw color will be obtained.

#### Test for the Purity of Rhubarb.

(1) Moderately heat 0.1 gm. ( $1\frac{1}{2}$  grs.) of the powdered Rhubarb with 20 drops of Olive-oil for one minute. On placing 1 drop of the mixture on a piece of white filter papering, a characteristic yellow ring will denote the presence of curcuma. If the Rhubarb is pure, the ring will have a scarcely perceptible yellowish tint.

#### Test for the Purity of Saffron.

(1) Concentrated sulfuric acid (Reagent 21) is the surest means of testing Saffron. The stigmas of the genuine article will immediately assume an indigo color, which changes quickly into dark red and brown, while the leaves of *Crocus vernus*, the most common adulteration of Saffron, are colored dark green.

#### Test for the Purity of Soap.

(1) Carefully weigh a piece of the Soap to be tested, cut it into thin chips or slices, then place it into soft water to which has been added a handful of table salt, set the pot on a slow fire until it comes to a boil. Keep boiling until all the Soap is dissolved, then set away to cool. The Soap eventually separates from the water, is then collected, allowed to dry, and reweighed. The loss in weight represents the amount of foreign matter present in the Soap.

### Tests for the Purity of Sugar.

Sugar is white, dry, hard, distinctly crystalline granules, odorless, and having a purely sweet taste. Permanent in the air. The aqueous solution, saturated at 25° C., has a specific gravity of about 1.340, is miscible with water in all proportions, and should be colorless. Soluble in 0.46 part of water, and in 137.2 parts of alcohol at 25° C.; in 0.2 parts of boiling water, and in 28 parts of boiling alcohol; also soluble in 80 parts of boiling absolute alcohol, but insoluble in ether, chloroform, or carbon disulfid. The aqueous or alcoholic solution of Sugar is neutral to litmus paper.

(1) Both the aqueous and the alcoholic solution of Sugar should be clear and transparent. When kept in large, well-closed, and completely filled bottles, the solutions should not deposit a sediment on prolonged standing (absence of *insoluble salts*, *ultramarine*, *Prussian blue*, etc.).

(2) If 1 gm. ( $15\frac{1}{2}$  grs.) of Sugar be dissolved in 10 c.c. (162 minims) of boiling water, the solution mixed with 4 or 5 drops of silver nitrate T.S., then about 2 c.c. (32 minims) of ammonia-water (Reagent 1) added, and the liquid quickly brought to the boiling-point, not more than a slight coloration, and no black precipitate, should appear in the liquid after standing at rest for five minutes (absence of *glucose*, and of more than a slight amount of *inverted sugar*).

(3) Cane-sugar may be distinguished from grape-sugar by Trommer's test, which consists in the use of copper sulfate and caustic potassa. If a solution of cane-sugar be mixed with a solution of copper sulfate, and potassa added in excess, a deep-blue liquid is obtained, which on being heated lets fall, after a time, a little red powder. A solution of grape-sugar, similarly treated, yields, by

heat, a copious greenish precipitate, which rapidly changes to scarlet and eventually to dark red.

(4) When a liquid containing grape-sugar is boiled with sodium carbonate and some basic bismuth nitrate, a gray coloration or blackening of reduced bismuth is produced. Cane-sugar, similarly treated, has no effect on the test.

(5) In carbon tetrachlorid, obtained by decomposing carbon disulfid by chlorin and aqueous vapor, a new test is found for distinguishing glucose from cane-sugar. This test, mixed with cane-sugar in a test-tube, kept for some time near  $100^{\circ}\text{C}.$ , causes a darkening till it becomes black. Glucose undergoes no such change.

### Test for the Purity of Sulfonal.

(1) Pure Sulfonal should be perfectly white and inodorous, it should have no appreciable bitter taste, its melting-point should be  $125.5^{\circ}\text{C}.$ , and should not deviate from this by more than  $0.2^{\circ}\text{C}.$  A saturated aqueous solution should be without effect at ordinary temperature on a  $\frac{1}{10}\%$  solution of potassium permanganate; and when 1 gm. ( $15\frac{1}{2}$  grs.) is boiled in 10 c.c. (162 minims) of water, no odor should be produced; if there be any, it is probably due to mercaptol.

### Test for the Purity of Tragacanth.

(1) A 1:30 solution of the suspected gum, made without heat, is mixed with an equal volume of a 1:100 aqueous solution of guaiacol; a drop of hydrogen peroxid is then added, and the mixture shaken. If acacia is present, the mixture rapidly acquires a brown color, whereas if the Tragacanth is pure, the mixture remains colorless.

### Test for the Purity of Turpentine.

(1) Drop a small quantity on a piece of white paper, expose it to the air, and if the Turpentine is pure, no traces will be left. If oil or other foreign matter is present, the paper will be greasy or soiled.

(2) Put 10 drops of the suspected Turpentine on a slightly concave watch-glass, allowing the glass to swim upon boiling water. Pure Turpentine should completely evaporate after seven minutes, while, if petroleum be present, the latter will remain; the percentage can be ascertained by weighing the glass and contents before and after the operation.

(3) Have a porcelain dish filled with water placed on a steam-chest or steam-bath, where the temperature of the water can be maintained at  $133.3^{\circ}\text{C.}$ , and put into this water two small porcelain or glass dishes of the same form, previously weighed, and containing each a certain weighed portion of the suspected article and of pure Turpentine. When the latter has completely evaporated, reweigh the dishes, and the difference, if any, will give the amount of adulteration within one-tenth part.

(4) Pure Turpentine has a specific gravity of 0.864 at  $15.5^{\circ}\text{C.}$ , and weighs 7 pounds and  $3\frac{1}{2}$  ounces to the gallon, and boils between  $156^{\circ}$  and  $162^{\circ}\text{C.}$

(5) Take two test-tubes, designate them as No. 1 and No. 2. In tube No. 1 place a sample of Turpentine known to be pure; also place some of this same Turpentine in tube No. 2, and add to it a small quantity of naphtha. Now, by placing a small crystal of potassium iodid in each tube, in twenty-four hours it will be observed that the chemical will have remained perfectly white and therefore unchanged, with regard to tube No. 1, in which the pure Turpentine was placed, while in tube

No. 2, in which the naphtha was associated with the pure Turpentine, the chemical will be found to have changed to a yellow color.,

(6) Fill two large test-tubes, one with a sample of Turpentine known to be pure, and the other with the sample to be tested. If mineral-oil be present in the suspected sample, by placing the tubes over a black cloth, that bluish-yellow bloom characteristic of the products of petroleum becomes at once apparent.

(7) The presence of adulterants in a suspected sample of Turpentine may be ascertained by violently shaking the bottle. If pure, the bubbles caused by the agitation will instantly disappear; but if, on the contrary, their disappearance be slow, adulterants are present.

### Test for the Purity of Vanilla.

(1) A small quantity of alcoholic solution of phloroglucin is placed in a watch-glass, and an equal volume of hydrochloric acid (Reagent 5) added. A crystal of the suspected substance is then added by the aid of a needle, when a magnificent red coloration is produced if it is Vanillin, whilst no color appears if the crystals consist of benzoic acid.

(2) To a solution supposed to contain Vanillin add a few drops of a 1 per cent solution of ferrous sulfate, and then bromin-water (Reagent 2), drop by drop. Vanillin gives a bluish-green color, turning yellow after a time.

### Test for the Purity of Vermillion.

(1) Mix the dry powder with sulfuric ether in a test-tube, shake thoroughly and filter. If only very minute portions of eosin or foreign coloring-matter be present, it will show a discoloration of the filtrate.

### Test for the Purity of Vinegar.

Vinegar is a sour liquid, the product of the acetous fermentation. Viewed chemically, it is a very dilute solution of acetic acid ( $\text{HC}_2\text{H}_3\text{O}_2$ ) containing certain foreign matters. When good is of an agreeable, penetrating odor and a pleasant acid taste. The color of Vinegar varies from pale yellow to deep red. When long kept, especially if exposed to the air, it becomes ropy, acquires an unpleasant smell, putrefies, and loses its acidity. It should be of such a strength that one fluid-ounce would require, for saturation, not less than 2.25 gm. (35 grs.) of potassium bicarbonate.

(1) Put 30 c.c. (1 f. oz.) of the suspected Vinegar into a small porcelain capsule over a water-bath, and evaporate to about 2 c.c. (32 minims), or to the consistency of a thin extract. When cool, add 15 c.c. ( $\frac{1}{2}$  f. oz.) of stronger alcohol, and thoroughly triturate. The free sulfuric acid, if present, will be taken up by the alcohol to the exclusion of any sulfate. Filter the alcoholic solution, add 30 c.c. (1 f. oz.) of distilled water, evaporate off the alcohol, and filter. Acidulate the filtrate with hydrochloric acid (Reagent 5), add a few drops of a solution of barium chlorid, and a white precipitate of barium sulfate will result, if the sample of Vinegar has been adulterated with sulfuric acid.

(2) Evaporate a sample of Vinegar in contact with a piece of white sugar or on white paper, if it contains free sulfuric acid the sugar or paper will be charred.

(3) Prepare a solution of logwood from boiling water and fresh logwood chips. Separate drops of this solution are spotted on the surface of a flat porcelain dish and evaporated to dryness over a water-bath. To each spot a drop of the suspected sample (concentrated first

if desirable) is added, and the heating continued until it has evaporated. If the Vinegar be pure, the residue will be found to have a bright-yellow color, but in the presence of a very small proportion of mineral acid the residue assumes a red color.

(4) Evaporate the Vinegar gently to an extract, and taste it; it will have an acrid, biting taste if red pepper, pellitory, grains of paradise, or mustard-seed is the adulterant.

(5) Copper may be detected by a brownish precipitate on the addition of potassium ferrocyanid to the concentrated Vinegar.

(6) Lead may be detected by the formation of a blackish precipitate on the addition of hydrogen sulfid, or by a yellow precipitate with potassium iodid.

(7) Lead is present when a sample of Vinegar is evaporated to one-quarter of its volume, and this yields a white precipitate when treated with sulfuric acid.

(8) Evaporate the Vinegar nearly to dryness. The residue is extracted with alcohol, filtered, and treated with a solution of potassium chlorid. A white precipitate indicates adulteration with tartaric acid.

(9) Mix a sample of the Vinegar with solution of sulfate of indigo. The fluid becomes discolored or assumes a yellowish color if adulterated with nitric acid.

(10) Add to a sample of Vinegar some powdered starch, boil it for half an hour, then allow it to become entirely cold (this is absolutely necessary), and add a few drops of iodine solution. The Vinegar, if adulterated with sulfuric acid, will be colored blue.

(11) Evaporate the Vinegar to one-fourth its volume and add ammonia-water (Reagent 1) in excess; a blue color indicates the presence of copper.

(12) Put a wineglassful of the Vinegar into a china teacup, and let the cup float in water in a pint cup of

tin or other metal that will stand heat. Boil the water till half the Vinegar has evaporated, then drop into the cup a piece of (cane) loaf-sugar about the size of a grain of wheat. Continue the boiling till the liquid in the cup has evaporated, when, if the Vinegar contains free sulfuric acid, the dry residue will be found to be blackened.

### Test for the Purity of Water.

Pure and wholesome Water must be limpid, odorless, transparent, cool, well aerated, bright, soft, fresh, tasteless, and entirely free from suspended matter of any sort, and perfectly neutral to litmus paper.

(1) On evaporating 1000 c.c. (34 f. oz.) of Water on a water-bath, it should not leave a residue weighing more than 0.5 gm. (7½ grs.) (limit of soluble salts), and this residue when ignited should not carbonize nor evolve *ammoniacal* or *acid* vapors.

(2) If 200 c.c. (6 f. oz., 366 minims) of Water be acidulated with hydrochloric acid (Reagent 5), heated to boiling, and 0.5 c.c. (8 minims) of barium chlorid T.S. be added, the liquid, when cooled and filtered, should give no further precipitate on the addition of a few drops of barium chlorid T.S., even on standing (limit of *sulfates*).

(3) If 200 c.c. (6 f. oz., 366 minims) of Water be acidulated with nitric acid (Reagent 15), heated to boiling, and 0.5 c.c. (8 minims) of tenth-normal silver nitrate V.S. be added, the liquid, when cooled and filtered, should not be affected by the subsequent addition of a few drops of silver nitrate T.S. (limit of *chlorids*).

(4) If 10 c.c. (162 minims) of Water mixed with a few drops of diphenylamin T.S. be carefully poured upon about 3 c.c. (48 minims) of sulfuric acid (Reagent 21), free from nitrous compounds, contained in a test-tube so as to form a separate layer, no blue color should be

found at the line of contact of the two liquids (limit of *nitrates*).

(5) If to 50 c.c. (1 f. oz., 331 minims) of Water contained in a glass cylinder 2 c.c. (32 minims) each of sulphanilic acid T.S. and naphthylamin acetate T.S. are added, and the solution well mixed, no distinct pink coloration should appear within five minutes, if the cylinder be placed upon a white surface and viewed from above (limit of *nitrites*).

(6) If to 50 c.c. (1 f. oz., 331 minims) of Water contained in a glass cylinder 2 c.c. (32 minims) of alkaline mercuric potassium iodid T.S. (Nessler's Reagent, see Ammonia (6)) be added and thoroughly mixed, no yellow or brownish tint should be produced immediately; the cylinder should be placed upon a white surface and viewed from above (limit of *ammonia*).

(7) On heating to boiling 100 c.c. (3 f. oz., 183 minims) of Water acidulated with 10 c.c. (162 minims) of diluted sulfuric acid, and subsequently adding 0.4 c.c. (7 minims) of tenth-normal potassium permanganate V.S., the pink color of the liquid should not be completely destroyed after it has been boiled for ten minutes (limit of *organic or other oxidizable substances*).

(8) Evaporate by gentle heat a small sample of the Water nearly to dryness in a clean porcelain cup, moisten the residue with acetic acid, and add to a portion of it a few drops of strong hydrosulfuric acid; a black precipitate indicates *lead*.

(9) Add to another portion of the residue with acetic acid a little pure hydrochloric acid (Reagent 5); a white precipitate, which redissolves on diluting with boiling Water, indicates *lead*.

(10) To the remainder of the solution of the residue in acetic acid add a few drops of dilute sulfuric acid and let it stand for a time; a white, heavy precipitate indicates *lead*.

(11) Dissolve a small quantity of good soap in alcohol. Let a few drops fall into a glass of the Water. If it turns milky, it is *hard*; if not, it is *soft*.

(12) Take litmus paper dipped in vinegar, and if, on immersion in the Water, the paper returns to its true shade, the Water does not contain *earthy matter* or *alkali*.

(13) If a few drops of syrup be added to a Water containing an *earthy matter*, it will turn green.

(14) Take equal parts of the Water and clear lime-water, and mix them. If combined as free *carbonic acid* is present, a precipitate is seen, to which, if a few drops of hydrochloric acid (Reagent 5) be added, an effervescence commences.

(15) Boil the Water to a twentieth part of its weight, and then drop a few grains of neutral carbonate of ammonia into a glass of it, and a few drops of phosphate of soda solution. If *magnesia* be present, it will fall to the bottom.

(16) Boil a little nutgall and add it to the Water. If it turns gray or slate-black, *iron* is present.

(17) Dissolve a little prussiate of potash in the Water, and, if *iron* is present, it will turn blue.

(18) Into a glass of the Water put 2 drops of a solution of oxalic acid and blow upon it. If it becomes milky, *lime* is present.

(19) If a piece of blue sugar paper immersed in the Water turns red, the Water contains a *mineral acid*.

(20) If *organic matter* be present, the Water becomes dark-colored by exposure to light.

(21) An ounce or two of the Water may be placed in a glass or porcelain vessel, to which may be added several drops of chlorid of gold solution sufficient to render the Water of a yellow tinge. If the Water contains no undue amount of *organic matter*, boiling it will not affect the yellow tinge, but if this matter be present in too great a

proportion, the fluid will assume a brown color, and then a violet or blue.

(22) *Organic matter* may be detected by evaporating the Water to dryness in a glass tube and then igniting, when there will be a smoke, odor, and charring.

(23) A few drops of lead-acetate solution added to the Water should produce only a faint turbidity (limit of *carbonic acid*).

(24) When the Water is mixed with an equal volume of calcium-hydrate solution, no cloudiness should appear (absence of *carbonic acid*).

### Test for the Purity of White Lead Paint.

(1) A dessert-spoonful of the paint is put in a bottle, and deprived of its oil by shaking it with gasoline. The mixture is then poured upon white filter-paper and the gasoline drained off. The powder is now put in another bottle and shaken with some vinegar. If after a minute or so it all dissolves, the paint is pure White Lead. If some remain undissolved, it may be because there was insufficient acid to combine with the lead, and more vinegar should be added. If there be still a residue, the paint has been adulterated.

(2) For dry White Lead take a test-tube, thoroughly dry and clean, and place therein about 20 grs. of the suspected article, and then fill the tube half full with dilute nitric acid. Pure White Lead will show effervescence and dissolve completely in a short time, while barytes will remain as an insoluble precipitate in the bottom of the tube.

(3) For White Lead in oil the blowpipe-flame test is the most simple. A small piece of the White Lead is placed in a cavity of a piece of charcoal under the steady flame of a blowpipe. Pure White Lead will be readily reduced

to a button of metallic lead, leaving the charcoal without a trace of any other substance. If zinc barytes, whiting, clay, or silica be present only to the extent of 5 per cent, there will be no formation of the metallic button, but the substance will have the appearance of a whitish-yellow or gray, cinder-like mass. Sulfate of lead can be reduced only with difficulty after long-continued blast or with the addition of powdered borax as a fusing material.





## LIST OF PRINCIPAL REAGENTS USED IN THIS BOOK

- 1 AMMONIA-WATER.—Sp. gr.  $0.96 = 10\%$   $\text{NH}_3$ .
- 2 BROMIN-WATER.—Dissolve 1 gm. ( $15\frac{1}{2}$  grs.) of Br in 30 c.c. (1 f. oz.) of water.
- 3 CHLORIN-WATER.—Should have a green tinge and be kept in a glass-stoppered bottle, standing upside down, in a dark place; it will keep, if thus preserved, for a long time.
- 4 FERRIC-CHLORID SOLUTION.—This solution must be free from uncombined HCl. Sp. gr. 1.281. Dissolve 29 gm. (1 oz., 10 grs.) of  $\text{Fe}_2\text{Cl}_6$  in 71 c.c. (2 f. oz., 192 minims) of water.
- 5 HYDROCHLORIC ACID CP.—Sp. gr.  $1.124 = 25$  HCl in 100.
- 6 IODIN-WATER.—A saturated watery solution of about 1 gm. ( $15\frac{1}{2}$  grs.) of I in 4 litres (135 f. oz.) of water.
- 7 IODIN, 0.5 gm. (7.7 grs.) AND POTASSIUM IODID.—1.5 gm. (23 grs.) in 250 c.c. ( $8\frac{1}{2}$  f. oz.) of water.
- 8 KALIUM BROMATE ( $\text{KBrO}_3$ ).—POTASSIUM BROMATE.
- 9 KALIUM IODATE ( $\text{KIO}_3$ ).—POTASSIUM IODATE.
- 10 LIME-WATER.—A saturated solution of calcium hydrate; contains about 1 gm. ( $15\frac{1}{2}$  grs.)  $\frac{1}{2}$ CaO to the litre (34 f. oz.).
- 11 MERCURIC CYANID.—Dissolve 5 gm. (77 grs.) of  $\text{Hg}(\text{CN})_2$  in 30 c.c. (1 f. oz.) of water.
- 12 MERCURIC-POTASSIUM-IODID SOLUTION  $\text{HgI}_2(\text{KI})_2$ .—Dissolve 22.70 gm. (351 grs.)  $\text{HgI}_2$  and 16.60 gm. (256 grs.) KI in one litre (34 f. oz.) of water; keep a certain quantity of a solution of  $\frac{1}{10}$  of this strength.
- 13 MERCURIC-CHLORID SOLUTION,  $5\%$ .—Dissolve 1 gm. ( $15\frac{1}{2}$  grs.)  $\text{HgCl}_2$  in 19 c.c. (308 minims) of water.
- 14 MERCURIC-BROMIDE SOLUTION: about  $0.5\%$ .—Dissolve 1 gm. ( $15\frac{1}{2}$  grs.)  $\text{HgBr}_2$  in 215 c.c. (7 f. oz., 129 minims) of water.
- 15 NITRIC ACID CP.—Sp. gr.  $1.185 = 30$   $\text{HNO}_3$  in 100.
- 16 PICRIC ACID; SATURATED SOLUTION.—Dissolve 10 gm.

- (154 grs.) pure crystals of Trinitrophenol ( $\text{C}_6\text{H}_2(\text{NO}_2)_3\text{OH}$ ) in 90 c.c. (3 f. oz., 20 minims) of water.
- 17 POTASSIUM-DICHROMATE SOLUTION, 5%.—Dissolve 2.5 gm. (39 grs.) of pure  $\text{Cr}_2\text{O}_7\text{K}_2$  in 47.5 c.c. (1 f. oz., 282 minims) of water.
  - 18 POTASSIUM-FERRICYANID SOLUTION, 5%.—To be made fresh when wanted by dissolving 1 gm. ( $15\frac{1}{2}$  grs.)  $\text{K}_3\text{Fe}(\text{CN})_6$  in 19 c.c. (308 minims) of water.
  - 19 POTASSIUM-FERROCYANID SOLUTION, 5%.—Dissolve 2.50 gm. (38 grs.)  $\text{K}_4\text{Fe}(\text{CN})_6 + 3\text{H}_2\text{O}$  in 47.5 c.c. (1 f. oz., 282 minims) of water.
  - 20 SODIUM-HYDRATE SOLUTION of 1.17 sp. gr. = 15% NaOH.—Do not make any larger quantity than 250 c.c. (8 f. oz., 217 minims). Dissolve 35 gm. (539 grs.) of NaOH from sodium in 250 c.c. (8 f. oz., 217 minims) of water and lubricate the glass stopper with vaseline.
  - 21 SULFURIC-ACID PURISSIMUM, sp. gr. 1.840; contains about 97%  $\text{H}_2\text{SO}_4$ .—Must not become colored with brucin, codein, morphin, or papaverin; it is very difficult to obtain an acid of such purity, which fact must be taken into consideration when a color-reaction appears with the acid. Compare the articles named under each one's separate heading.
  - 22 SULFURIC ACID CONTAINING CHROMIC ACID.—Dissolve 0.02 gm. ( $\frac{5}{16}$  gr.) of  $\text{Cr}_2\text{O}_7\text{K}_2$  in 10 c.c. (162 minims) of water and add to the solution 30 gm. (465 grs.) of concentrated  $\text{H}_2\text{SO}_4$ . To be made fresh when wanted.
  - 23 SULFURIC ACID CONTAINING POTASSIUM PERMANGANATE.—Dissolve 0.02 gm. ( $\frac{5}{16}$  gr.)  $\text{MnO}_4\text{K}$  in 10 c.c. (162 minims) of water and add 30 gm. (465 grs.) of concentrated  $\text{H}_2\text{SO}_4$  to the solution. To be made fresh when wanted.
  - 24 TANNIN SOLUTION.—To be made fresh when wanted by dissolving 1 gm. ( $15\frac{1}{2}$  grs.) of tannin in 19 c.c. (308 minims) of water.
  - 25 SULFURIC ACID CONTAINING SODIUM MOLYBDATE.—Dissolve 0.1 gm. ( $1\frac{1}{2}$  grs.) sodium molybdate in 10 c.c. (162 minims strong  $\text{H}_2\text{SO}_4$  (Fröhde's tests).

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